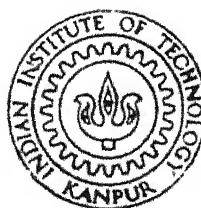


 **STUDIES ON HOT AND COLD ROLLING OF POROUS STRIPS
MADE FROM ATOMIZED COPPER POWDER**

By
K. JANAKI KODANDA RAM

TH
ME/1991/M
R1458



**DEPARTMENT OF METALLURGICAL ENGINEERING
INDIAN INSTITUTE OF TECHNOLOGY KANPUR
APRIL, 1991**

STUDIES ON HOT AND COLD ROLLING OF POROUS STRIPS MADE FROM ATOMIZED COPPER POWDER

*A Thesis Submitted
in Partial Fulfilment of the Requirements
for the Degree of
MASTER OF TECHNOLOGY*

By
K. JANAKI KODANDA RAM

to the

**DEPARTMENT OF METALLURGICAL ENGINEERING
INDIAN INSTITUTE OF TECHNOLOGY KANPUR
APRIL, 1991**

112178

ME-1991-M-RAM-STU

CERTIFICATE

DATE OF i
S. Bhargava
Submitted on 16/4/91
R. K. Dube

This is to certify that the thesis entitled "Studies on hot and cold rolling of porous strips made from atomized copper powder" submitted by Mr. K.J. Kodanda Ram has been carried out under my supervision and has not been submitted elsewhere for a degree.

S. Bhargava

(S. Bhargava)
Assistant Professor
Dept. of Metallurgical Engg.
Indian Institute of Technology,
Kanpur

R. K. Dube

(R. K. Dube)
Professor
Dept. of Metallurgical Engg.
Indian Institute of Technology,
Kanpur

ACKNOWLEDGEMENTS

At the very onset I wish to express my earnest respect and gratitude to my thesis supervisors for their invaluable guidance, constant encouragement and endless cooperation throughout my tenure without which this investigation would never have been materialized successfully. The readymade solutions for all practical problems and the guidance to develop genuine experimental skill by Dr. R.K.Dube and Dr. S. Bhargava have ingratiated me beyond expression.

I must offer my sincere thanks to all my friends for their invaluable companionship and discussions during my stay at I.I.T. Kanpur. But special mention should be made to Mr. P. Umakanth, Mr. B.V.S. Perraju and Mr. C.N. Shanker for their constant cooperation in all stages of the laboratory work.

I convey my sincere thanks to Mr. S.C. Soni for his companionship for printing the microstructures and constant help during my stay at I.I.T. Kanpur.

I acknowledge with grateful thanks for the invaluable cooperation and help which I have received from the staff members, Engg. Metallurgy Lab. and ACMS.

In the end I wish to put on record my thanks to Mr. A.K. Srivastava for his flawless typing.

LIST OF FIGURES

FIGURE

1.1 Schematic flow diagram of P/M routes for making metal strip.

1.2 Schematic view of Direct cold metal powder rolling.

3.1 Schematic flow chart for the production of copper strip by slurry casting/Bonded metal powder rolling route.

3.2 Morphology of Atomized copper powder particles as examined by scanning electron microscope.

3.3 Views of sintered copper strip under the scanning electron microscope.

3.4 Schematic view of the set up used for the hot rolling of porous copper strips.

3.5 Tensile Test specimen.

4.1 Effect of hot rolling temperature and thickness reduction on the relative density of porous copper strip.

4.2 Effect of thickness reduction on inter connected and total porosities of hot rolled strip rolled at 1023K.

4.3 Effect of thickness reduction on interconnected and total porosities of hot rolled strip rolled at 1223K.

4.4 Schematic view showing three stages of densification of porous copper strip during its hot rolling.

4.5 SEM view showing particle arrangement at different stages of densification of porous copper strip during its hot rolling.

4.6 Optical micrographs in thickness direction of strips hot rolled at different temperatures and quenched.

4.7 Optical micrographs in thickness direction of strips hot rolled at different temperatures and cooled in graphite chips.

4.8 Inclusion size distribution histogram for copper strip prepared from atomized copper powder by hot rolling at 1223K and annealed at 823K.

4.9 Effect of hot rolling temperature on the Aspect ratio of grains, in as hot rolled copper strips.

4.10 Transmission electron micrographs showing the dislocation cell structure for fully dense hot rolled strips and quenched.

4.11 Transmission electron micrographs showing the dislocation cell structure for fully dense hot rolled strips and cooled in graphite chips.

4.12 Effect of hot rolling temperature on the tensile strength and 0.2% yield strength of strips hot rolled at different temperatures and quenched.

4.13 Effect of hot rolling temperature on the tensile strength and 0.2% yield strength of strips hot rolled at different temperatures and cooled in graphite chips.

4.14 Effect of hot rolling temperature on % Elongation of strips hot rolled at different temperatures and quenched.

4.15 Effect hot rolling temperature on % Elongation of strips hot roiled at different temperatures and cooled in graphite chips.

4.16 SEM view of Fractured surfaces after the tensile test on strips hot rolled at different temperatures and quenched.

4.17 SEM view of Fractured surfaces after the tensile test on strips hot rolled at different temperatures and cooled graphite chips.

4.18 Microstructure of copper strips hot rolled at 1223K and annealed at 823K.

4.19 SEM view of fractured surface after the tensile test on strip hot rolled at 1223K and annealed at 823K.

4.20 TEM view of strip hot rolled at 1223K and annealed at 823K.

4.21 SEM views of fully dense copper strip, prepared from atomized copper powder cold rolled to different deformations, showing decohesion and/or Fragmentation of Cu_2O inclusion.

4.22 Effect of cold rolling thickness reduction on Decohesion/Fragmentation of inclusions in strip prepared from atomized copper powder.

4.23 Inclusion size distribution histogram for copper strip prepared from cuprous oxide powder.

4.24 SEM views of fully dense copper strip, prepared from cuprous oxide powder cold rolled to different deformations, showing decohesion and/or Fragmentation of inclusions.

4.25 Effect of thickness reduction on UTS of copper strip cold rolled and annealed.

4.26 Effect of thickness reduction on % Elongation of copper strip cold rolled and annealed.

LIST OF TABLES

4.1 (a) Effect of thickness reduction on densification of Porous copper strip hot rolled at 1023K.

• (b) Effect of thickness reduction on densification of porous copper strip hot rolled at 1223K.

4.2 Effect of hot rolling temperature on the mechanical properties of strip hot rolled and quenched.

4.3 Effect of hot rolling temperature on the mechanical properties of strip hot rolled and cooled in graphite chips.

4.4 Effect of cold rolling thickness reduction on mechanical properties of strip cold rolled and annealed.

4.5 Effect of cold rolling thickness reduction on decohesion and/or fragmentation of Cu_2O inclusions.

1. INTRODUCTION

The traditional Powder Metallurgy (P.M.) processing consists of compacting metal powder into the required shape followed by sintering and post-sintering operation such as infiltration. The salient feature of such products is that it usually contains 3-5% porosity. The porosity may be even more depending upon the applications. However, a new class of material known as P.M. Wrought products, has been developed, which are produced by hot and cold working of powder preforms. They are characterised by the fact that they are fully dense and possess characteristic wrought microstructure. Metal strips can also be manufactured by Powder Metallurgy processing and is an example of the P.M. Wrought products.

1.1 CLASSIFICATION OF METHODS FOR STRIP PRODUCTION:

Conventional methods of strip production has many disadvantages as studied by earlier worker (2,3,5,6,7). Therefore there exists a need for developing alternative routes for thin strip production from an economically viable small sized plant with low capital investment. Out of the alternative routes continuous casting route and powder metallurgy route can be used for strip production. But powder metallurgy routes offer considerable advantages, both in terms of energy saving and low capital investment^(4,8). Therefore powder metallurgy methods can be used for production of strips.

There are several different P.M. routes ⁽⁹⁾ for making thin strips which has recently been reviewed by Dube ⁽¹⁰⁾. Fig 1.1 gives the schematic view of different processes for thin strip preparation. The entire processing of metal powder into finished strip by P/M routes based on cold metal powder rolling consists of following unit processes.

- (a) Green strip preparation
- (b) Sintering of Green strip
- (c) Densification rolling of Porous sintered strip
- (d) Final cold rolling and annealing.

The common factor in the above is densification rolling, since whatever the method of preparation may be there will be porosity in the strip after sintering and it is to be densified to get fully dense strip. Green strip preparation can be done either by (a) Direct cold metal powder rolling (b) Bonded metal powder/slurry casting route. Fig. 1.2 shows the schematic view of Direct cold metal Powder rolling.

1.2 DENSIFICATION ROLLING OF SINTERED METAL STRIP:

Sintered strips obtained from any of methods discussed above contain a considerable amount of porosity from 60% to 15%. The densification of the sintered strip into a fully dense strip can be carried out in two different ways:

- (1) Repeated cold rolling and annealing
- (2) Hot rolling

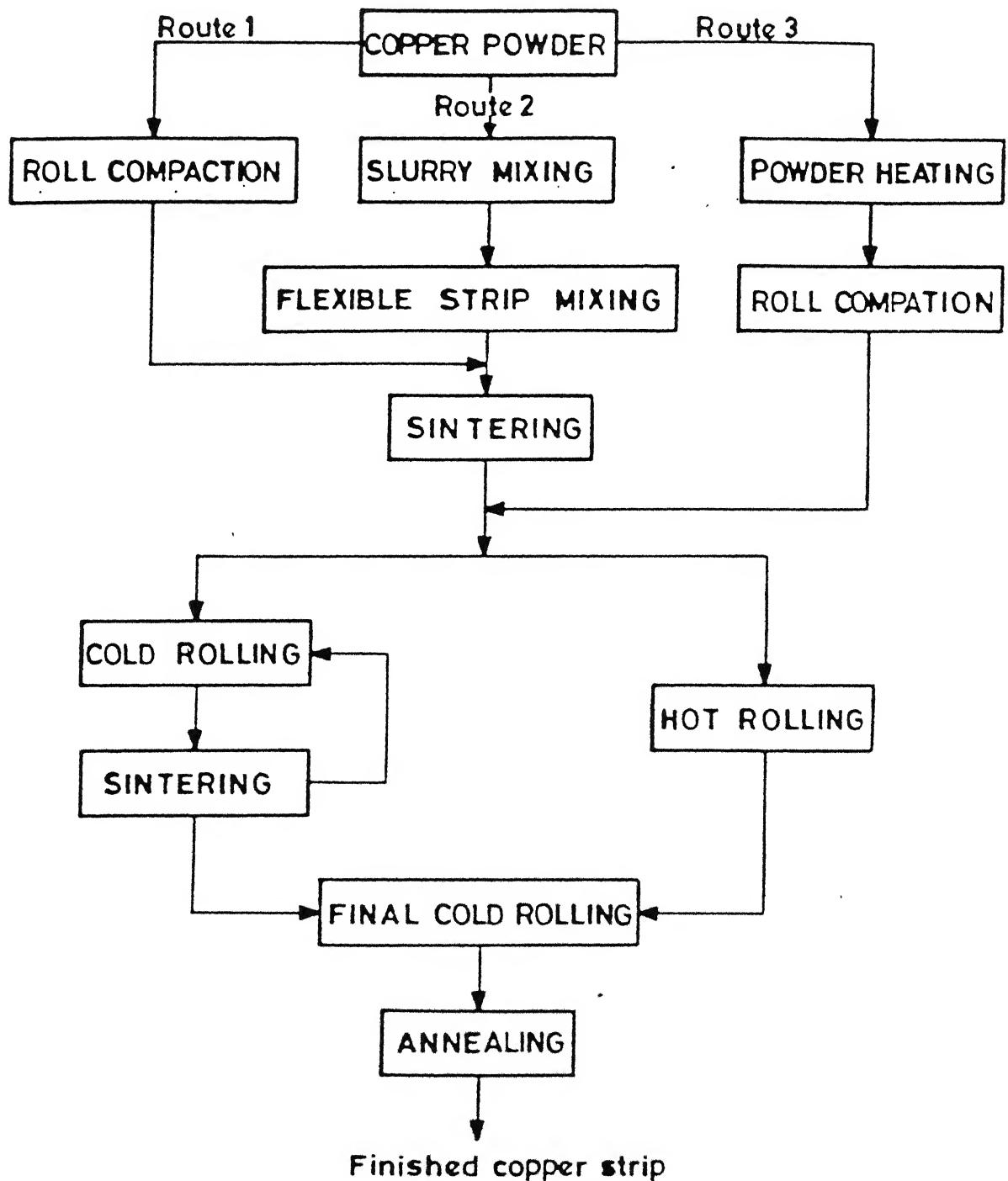


FIG.1.1 SCHEMATIC FLOW DIAGRAM OF P/M ROUTES FOR MAKING COPPER STRIP.

(Route 1 – Direct cold powder rolling,
 Route 2 – Bonded powder rolling ;
 Route 3 – Hot powder rolling).

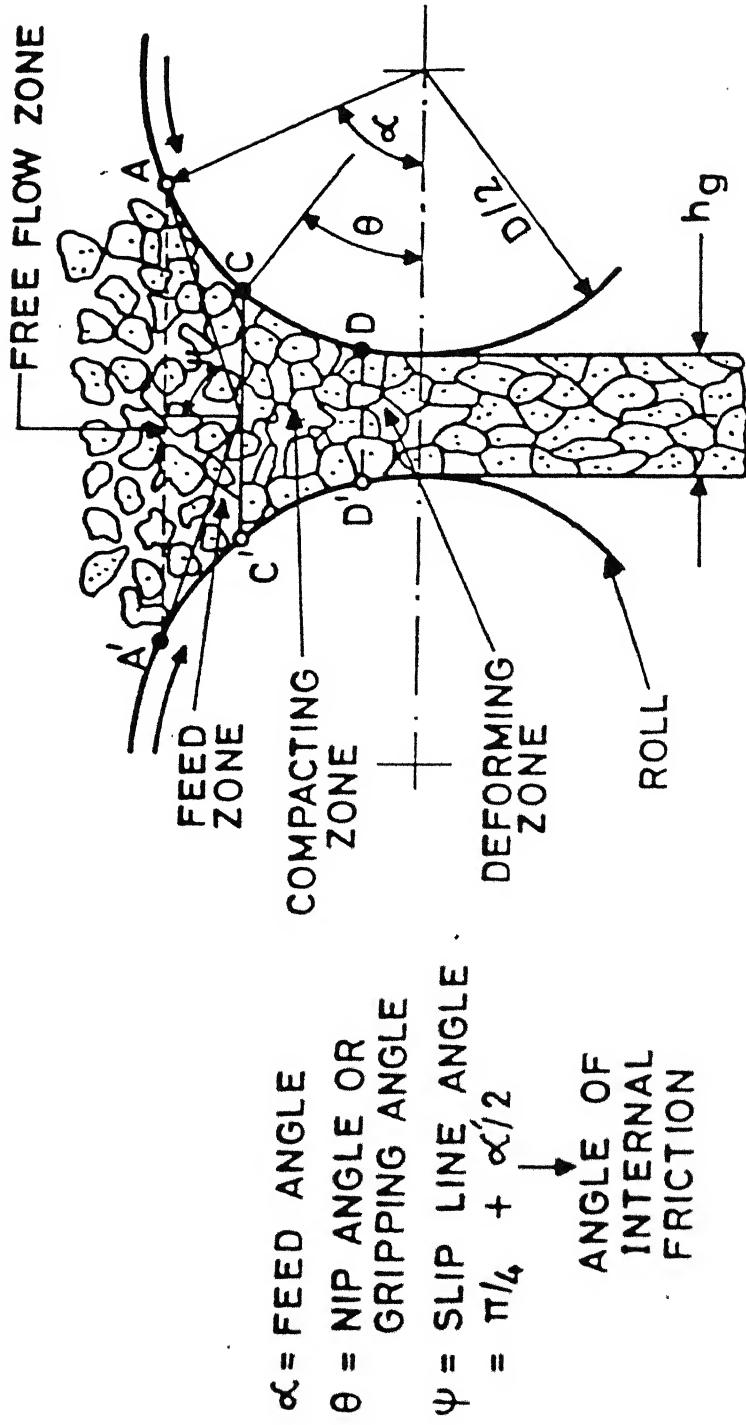


FIG.1.2 VARIOUS ZONES WITHIN THE ROLL BITE DURING THE
DIRECT COLD METAL POWDER ROLLING.

Variables in densification rolling can be categorized as below:

- (1) Powder characteristics
 - size
 - shape
 - distribution
- (2) Method of making Green strips
- (3) Density of sintered strip
- (4) Hot rolling temperature
- (5) Rolling mill parameters
 - (a) Diameter of rolls
 - (b) Speed of rolls
 - (c) Roll temperature
- (6) % Reduction
- (7) Rollability of metal

The effect of several variables on the feasibility and properties of strip rolled from powder routes has been studied by workers . However the effect of hot rolling temperature on the microstructural and mechanical properties has not been studied systematically. The present work has been undertaken to study the effect of hot rolling temperature on the microstructural and mechanical properties of the fully dense copper strips.

Many investigators⁽¹¹⁻¹³⁾ have favoured hot rolling after sintering as a means of achieving 100 percent density in the strip. The main advantages of this process are that it is flexible to produce a dense strip in a single operation, and it reduces the sintering time. Moreover it is possible to hot roll

the sintered strip directly from the furnace without cooling. The amount of thickness deformation required to produce a fully dense strip depends on the initial porosity and is always greater than that theoretically required. This is because some of the rolling deformation goes into elongating the strip instead of closing the pores. The experimental procedure for hot rolling of strip is described in chapter 3.

1.3 FURTHER COLD ROLLING AND ANNEALING OF HOT ROLLED STRIPS -

Sheet and strip metals are generally cold rolled, followed by a suitable anneal before the final finished product is shipped. This is done obviously to achieve optimum mechanical and structural properties in addition to superior surface finish. Powder metal strip produced by hot rolling route would have an inferior surface finish because of oxide film formation. Therefore it is necessary to pickle the strip/sheet before cold rolling. It is desirable to start final cold rolling on a fully dense P/M metal strip. However Smucker⁽⁴⁾ observed that copper strip with 96-100 percent density range could be cold rolled like a conventional 100 percent dense copper strip. This final cold rolling and annealing operations, therefore could be used for some densification. Nevertheless, the primary aim remains to achieve optimum mechanical properties.

Copper strip prepared from Atomized copper powder can contain non metallic inclusions. During deformation, due to the plasticity difference between matrix and inclusion, Decohesion or

Fragmentation of inclusion can take place. The effect of cold rolling of strips containing non metallic inclusion on mechanical and microstructural properties has not been studied systematically by earlier workers.

CHAPTER - 2

AIMS OF PRESENT INVESTIGATION

- (1) To study the strip densification with the percentage thickness reduction, and the effect of hot rolling temperature on the densification behaviour.
- (2) To study the various stages of densification using scanning electron microscope on strips hot rolled to different thickness reductions.
- (3) To study the effect of hot rolling temperature on the mechanical and microstructural properties of copper strips hot rolled at different temperatures and
 - (a) Quenched in water
 - (b) Cooled in graphite chips
- (4) To study the effect of annealing on the strip hot rolled at 1223K and annealed at 823K.
- (5) To study the quantitative analysis of inclusion in copper strips made from atomized copper powder.
- (6) To study the effect of cold rolling thickness reduction on mechanical and microstructural properties of strips cold rolled and annealed.

CHAPTER 3

EXPERIMENTAL PROCEDURES

Various powder metallurgy methods for the preparation of thin strips have been studied by many workers and it has recently been reviewed by Dube⁽¹⁰⁾. Strip preparation by bonded cold metal powder/slurry casting route method consists of preparation of slurry mixture from metal powder, a binder and a plasticizer mixed in a vehicle medium, casting and drying it to get a coherent strip. This strip obtained is then sintered and densified by rolling. The sequence of operations have been shown in Fig. 3.1.

3.1 MATERIALS :

3.1.1 Atomised copper powder:

The atomized copper powder for this study was supplied by Greenback Industries, Inc. U.S.A. The powder was 99.5 percent pure and had an apparent density of 2.83 mg/m^3 and standard Hall flowmeter rating of 34s/50g. Size distribution of the powder was as follows:

Mesh size	+ 100	+ 150	+ 200	+ 250	+ 325	- 325
Present	0.2	4.2	7.5	5.3	18.9	639

Examination of powder shape under scanning electron microscope revealed the spherical shape Figures 3.2, a & b, show the typical scanning photograph of the powder.

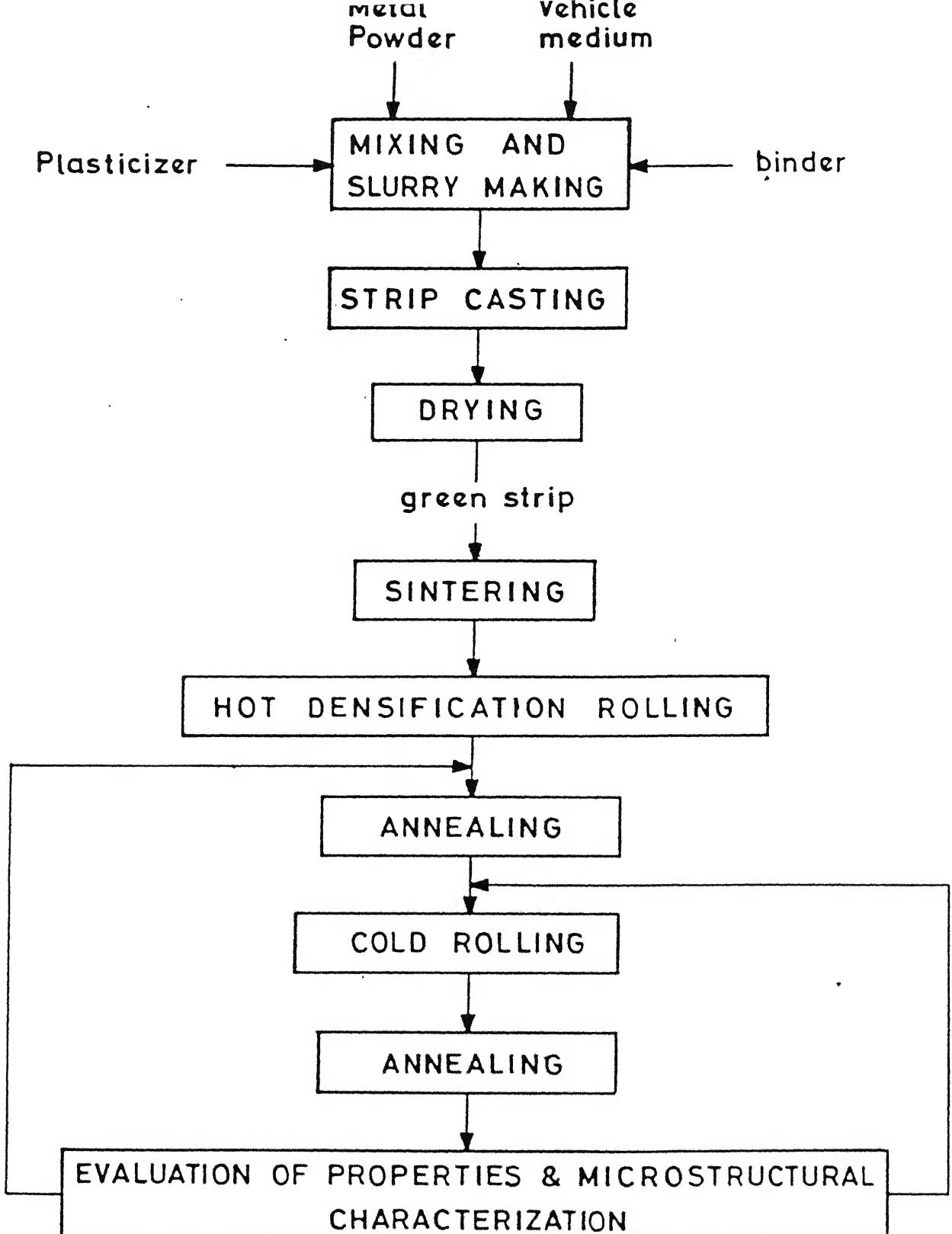
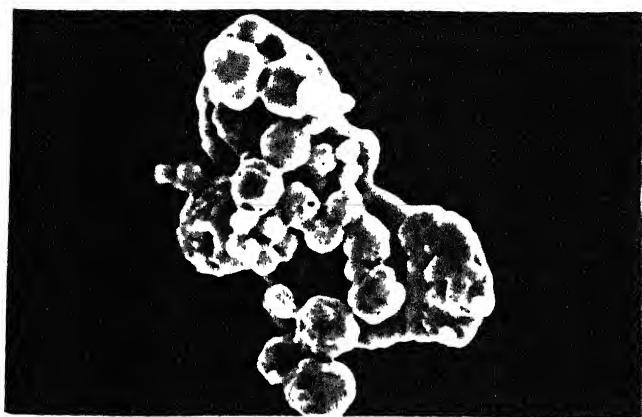
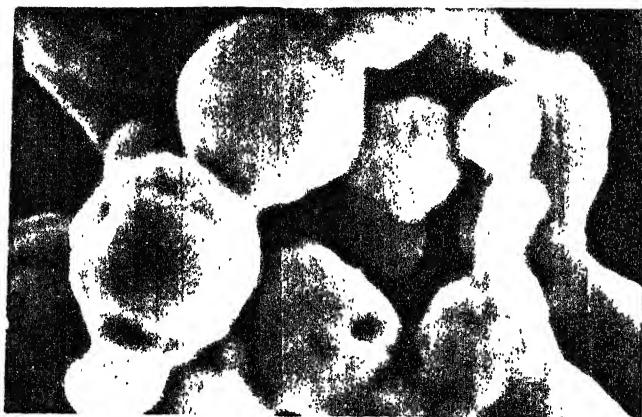


Fig.3.1 Schematic flow chart for the production and characterization of copper strips prepared by bonded metal powder/slurry casting route.



a (X3000)



b (X11000)

Fig. 3.2 Morphology of Atomized copper powder particles as examined by scanning electron microscope.

3.1.2 Binder

The purpose of binder is to provide sufficient strength to green dry strip after solvent is evaporated. Binders are basically film formers⁽¹⁵⁾ which are dissolved molecularly in the solvent or are dispersed in the liquid as an emulsion⁽¹⁶⁾. Organic binders are generally used for the preparation of strip. A binder, in general, should satisfy the following requirements:

- 1) It should form a solution of sufficient viscosity at low concentrations and should be available in a wide range of viscosity.
- 2) It must form a flexible self supporting film after drying
- 3) It should volatalize to a gas leaving negligible residual carbon or ash during sintering.
- 4) It should be relatively inexpensive.

Water soluble methyl cellulose [-CH₂-O-CH₃] was used as a binder in the present work.

3.1.3 Plasticizer:

Plasticizers are additives which soften the binder in the dry or near dry state. They are lower molecular weight organic compounds and dissolve on the same liquid as the binder. After drying the binder and plasticizer are intimately mixed as a single material. Plasticizer increases the flexibility of material⁽¹⁷⁾.

Methyl cellulose, when used as a binder, formed green

strip having a curled surface and was hard. According to Thomson⁽¹⁸⁾ Glycerene can be used as a plasticizer in a methyl cellulose - water system. Reagent grade glycerine was, therefore, used as a plasticizer.

3.1.4 Vehicle Medium:

A solvent or vehicle should have essentially the following characteristics: Low boiling point, low viscosity, ability to dissolve binder and plasticizer and non-reactivity with metal powder. Most of the non-aqueous solvents, eg: Acetone, Benzene etc offer the above advantages but are expensive as compared to that of water, also toxic in nature and can give rise to Flammability hazards⁽¹⁸⁾ water on the other hand is cheaper and non toxic but has relatively higher boiling point requiring extraneous heating. Water was, therefore used as a vehicle medium.

3.1.5 Gases :

Gases used in the present work are H₂, N₂ and Argon. Pure Hydrogen (IOLAR - 2 Grade) supplied by Indian Oxygen Ltd. was used as a protective atmosphere, during sintering of porous copper strip, to prevent oxidation of strip. Dry Nitrogen was used for flushing the chamber before passing Hydrogen in the furnace. Pure Argon was used for annealing purposes.

3.2 PREPARATION OF STRIP FROM ATOMIZED COPPER POWDER:

3.2.1 PREPARATION OF GREEN COPPER STRIP

The sequence of operations were given in Fig. 3.1 by trial and error, the following weight percent composition was found to behave the best for flow characteristics of the slurry and the strip produced from it is coherent.

Atomized copper powder	- 69.7%
Methyl cellulose	- 0.7 %
Glycerine	- 1.8 %
Water	- 27.8 %

In order to prevent the agglomerate formation during mixing of binder with solvent, dry premixing of binder with other ingredients is done and then slurry is prepared. The following methods be used to form the slurry.

- (a) Weighed quantities of Atomized copper powder and methyl cellulose are mechanically blended to form a uniform mixture.
- (b) Adequate quantities of water and Glycerine are mixed in a separate container so that they form another solution.
- (c) The solid and liquid constituents obtained from (a) and (b) are mixed in a beaker by means of laboratory stirrer for 15 minutes.

A homogeneous slurry resulted from the above procedure which had good flow characteristics. The slurry thus formed can contain some air bubbles which will resulted in small blow holes

and so care was taken to minimize the trapping of air bubbles in cast strip.

The slurry was poured into a flat horizontal steel mold of size $100 \times 75 \times 5$ mm. The slurry flowed on its own to fill the mould and the excess material was removed using a scraper. Before pouring the slurry the mould was coated with Oleic acid which acted as a releasing agent. The cast slurry was subsequently dried by heating the mould on a hot plate. In order to avoid excess heating the strip was removed from the mold when containing little amount of moisture and was further dried on hot air oven at 393°K . A coherent strip resulted after drying the green strip and a little shrinkage was observed.

The apparent density of the cast copper strip in dry condition was 2.205 mg/m^3 indicating that it contained about 75% porosity. The strip had enough strength for handling and did not pose any problem.

3.2.2 SINTERING OF GREEN COPPER STRIP:

The green copper strips were sintered in hydrogen atmosphere in a specially designed furnace. The heating chamber, which was heated by silicon carbide rods, consisted of an Inconel tube, 750 mm long and of 100 mm internal diameter and was closed from one end. The open end of the furnace had a 200 mm long cooling chamber where the atomized copper strips were coiled under hydrogen atmosphere. Gases were introduced into the chamber through a stainless steel tube passing through the open end of the

chamber.

The standard procedure for sintering was that the furnace chamber already maintained at the required temperature, was flushed with Nitrogen for 300s (5 min) before introducing hydrogen into it. The green strip, placed on a perforated inconel tray was introduced into the furnace chamber to the hot zone of the furnace.

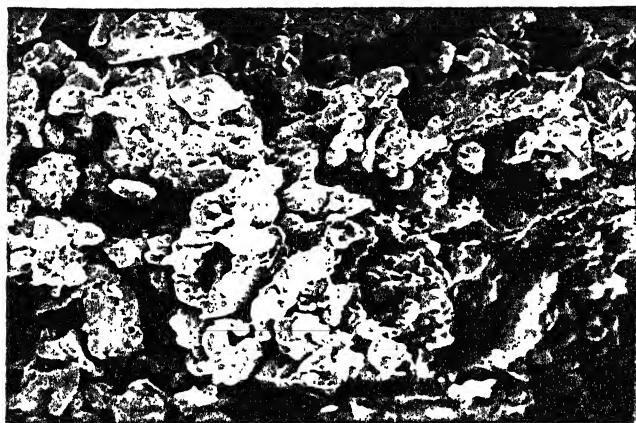
All the strips were sintered at 1223 K for 4500 sec in H_2 atmosphere and then cooled for 25 min in the cooling zone under hydrogen atmosphere. More time was given for sintering because, the powder was old and may have oxide coating over the particles. The cooled strips were taken out and were further densified. The typical density of the sintered strip was 3.2504 mg/m^3 (rel. density, 0.365) implying the porosity of ≈ 65 percent.

Fig 3.5 shows the view of the sintered copper strip showing inter connected porosity.

3.2.3 DENSIFICATION ROLLING OF POROUS COPPERSTRIP:

The copper strip obtained after sintering contained about 65% porosity. In order to obtain fully densified strip it is necessary to densify them by rolling, either cold or hot. In the present work the strips were hot rolled to get full density.

Four sets of strips were prepared upon which the following processing method has been adopted.



a (X270)



b (X1200)

Fig. 3.3 Vies of sintered copper strip under the scanning electron microscope.

(1) One set of copper strips were hot rolled to various thickness reductions giving partially densified strips. Two hot rolling temperatures were chosen for the above case, 1223K and 1023K, and the strips after hot rolling were cooled in graphite chips.

(2) Two sets of copper strips were hot rolled to full density in single pass at different temperatures. One of which were quenched in water after hot rolling and the for the other set, they were cooled in graphite chips. The temperature of hot rolling in this case were 923K, 1023K, 1123K and 1223K.

(3) One set of copper strips were hot rolled at 1223⁰K to full density in single pass and cooled in graphite chips and these were annealed and used for study of cold rolling behaviour.

3.2.4 HOT ROLLING OF POROUS COPPER STRIP :

The furnace used for reheating and hot rolling was same as the one described in section 3.3. Reheating and hot rolling was done in Hydrogen atmosphere to prevent oxidation of the strip. One end of reheating chamber was closed, while other end had a zone projecting outside the furnace, as shown in Fig. 3.4. The reheating chamber contained a flat inconel plate which acted as a base for the strip. Porous copper strips were preheated to corresponding temperature of hot rolling for 1800s (30 min) prior to hot rolling. The hot rolling was performed on a 2-high mill having 135 mm diameter rolls rotating at a speed of 55 rpm and maintained at room temperature. The required thickness

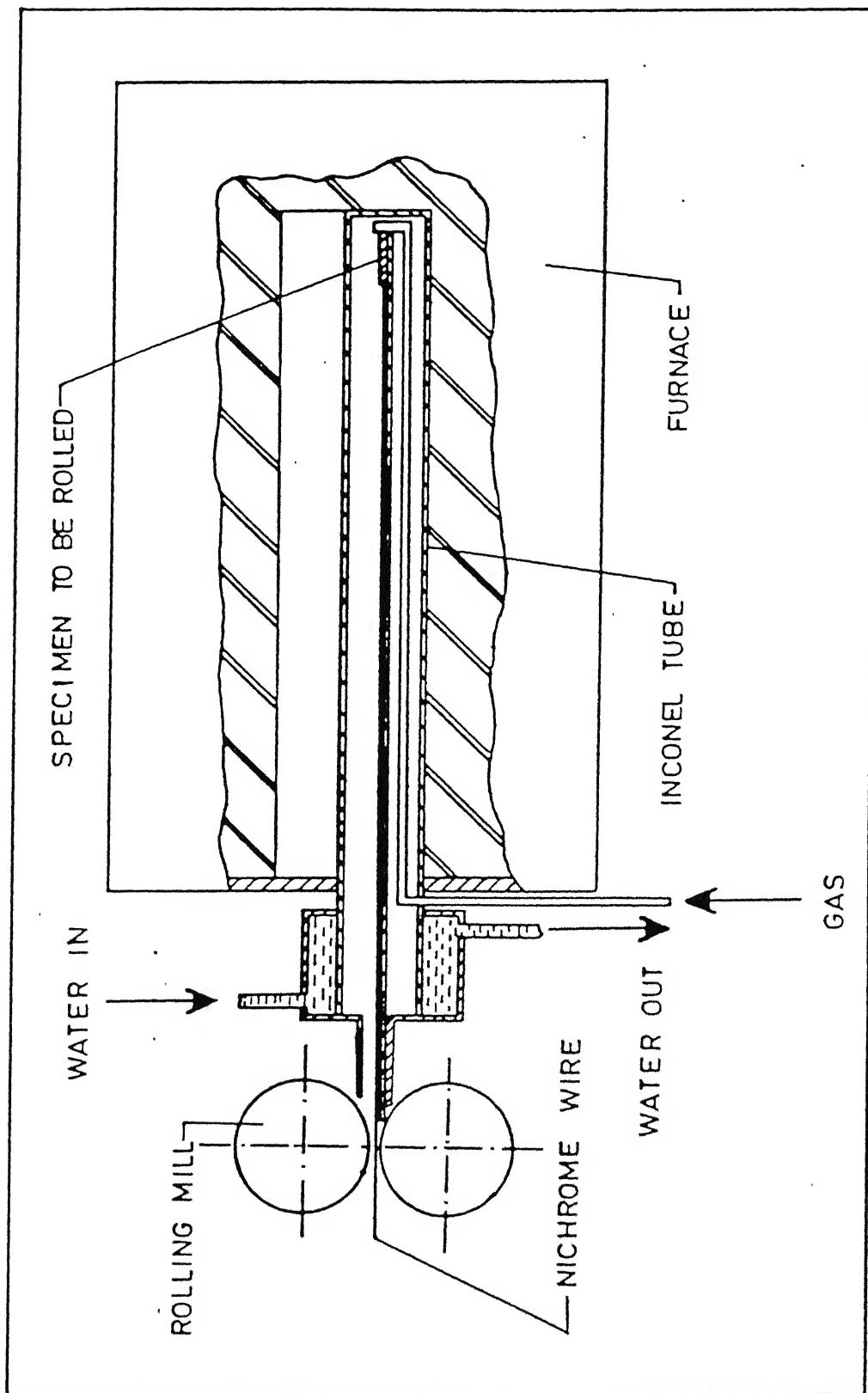


Fig.3.4 Hot rolling arrangement.

deformation was given by hot rolling in a single pass. In order to avoid internal oxidation of the strip via its inter connected porosity, hot rolling was carried out in such a way that it is under protective atmosphere upto very near the nip of rolls. The standard procedure for hot rolling was as follows:

- (1) A small hole was drilled near one edge of the porous copper stripd and a thin nichrome wire was attached to it.
- (2) An inconel plate was used for the introduction of the strip into the hot zone of the preheating furnace.
- (3) The preheating furnace, which is mobile, was placed in front of rolling mill so that the extended one at exit was very close to the nip of rolls.
- (4) The roll gap was adjusted to the required level and then the heated porous strip was pulled into the rolls, from the other side of rolls, using the attached wire.
- (5) The strips emerging out of the rolls were given the treatments as explained before in section 3.4.

3.3. COLD ROLLING OF HOT ROLLED STRIPS :

One set of strips hot rolled at 1223°K to full density in single pass, were colled in graphite chips after hot rolling. These strips were annealed at 823°K for 1800s in Argon atmosphere and colled for 1500s in colling zone. These were subjected to cold rolling to various thickness deformations. Cold rolling was performed on a 2 high mill. The direction of cold rolling was kept parallel to that of previous rolling directions.

3.4 METHODS OF TESTING AND INSPECTION

3.4.1 DENSITY MEASUREMENT:

The density of Green strip was calculated from weight and dimension measurements. Densities of sintered copper strips and further densified strips were determined by the immersion technique. Reagent grade Ethylene glycol was used as impregnating liquid. It has many advantages over other liquids. It has low vapour pressure, and therefore does start boiling when trapped air is pulled out from samples under vacuum. Also it can be removed completely by heating to a low temperature of 483°K (210°C) and is non-reactive towards copper.

The method consisted of immersing the samples in Ethyleneglycol and keeping whole arrangement in vacuum decicator. Due to the low pressure inside it enables the trapped air to come out from open pores. By repeating this process 3-4 times it was possible to remove all air from the samples. These open pores were then filled with Ethylene glycol. The samples were then wiped carefully to remove the glycol film on surface without pulling any glycol from the pores.

The impregnating step was eliminated where the specimens were known to be of higher density i.e. $> 95\%$ of theoretical density. Density interconnected, total and isolated porosities i.e. ρ_s : ϵ_{Inter} : ϵ_{Iso} respectively were calculated from following relation strips⁽¹⁹⁾:

$$\rho_s = \frac{\omega_1}{\omega_2 - \omega_3} \quad \dots \quad 3.1$$

$$\epsilon_{\text{inter}} = \frac{(\omega_2 - \omega_1) \times 100}{(\omega_2 - \omega_3)} \quad \dots \quad 3.2$$

$$\epsilon_{\text{total}} = \frac{\rho_t - \rho_s \times 100}{\rho_t} \quad \dots \quad 3.3$$

$$\epsilon_{\text{isolated}} = \epsilon_{\text{total}} - \epsilon_{\text{inter}} \quad \dots \quad 3.4$$

where ρ_s = density of the sample

ρ_t = density of copper taken to be 8.92 mg/m^3 during this study.

ω_1 = weight of the sample in air

ω_2 = weight of the sample in air after impregnation with glycol.

ω_3 = weight of the impregnated sample in water.

3.4.2 MECHANICAL PROPERTIES :

UTS, 0.2% proof stress, and percent elongation were determined by a universal testing machine, Instron 1150. The testing was done at room temperature and samples were strained at a rate of $4 \times 10^{-5}/\text{sec}$. Because of the shortage of strip material, the specimen used was not of standard specification⁽²⁰⁾. However, the geometry of the specimen, as shown in Fig. 3.5 was maintained according to the standard specification. A minimum of 2 samples were tested in each case.

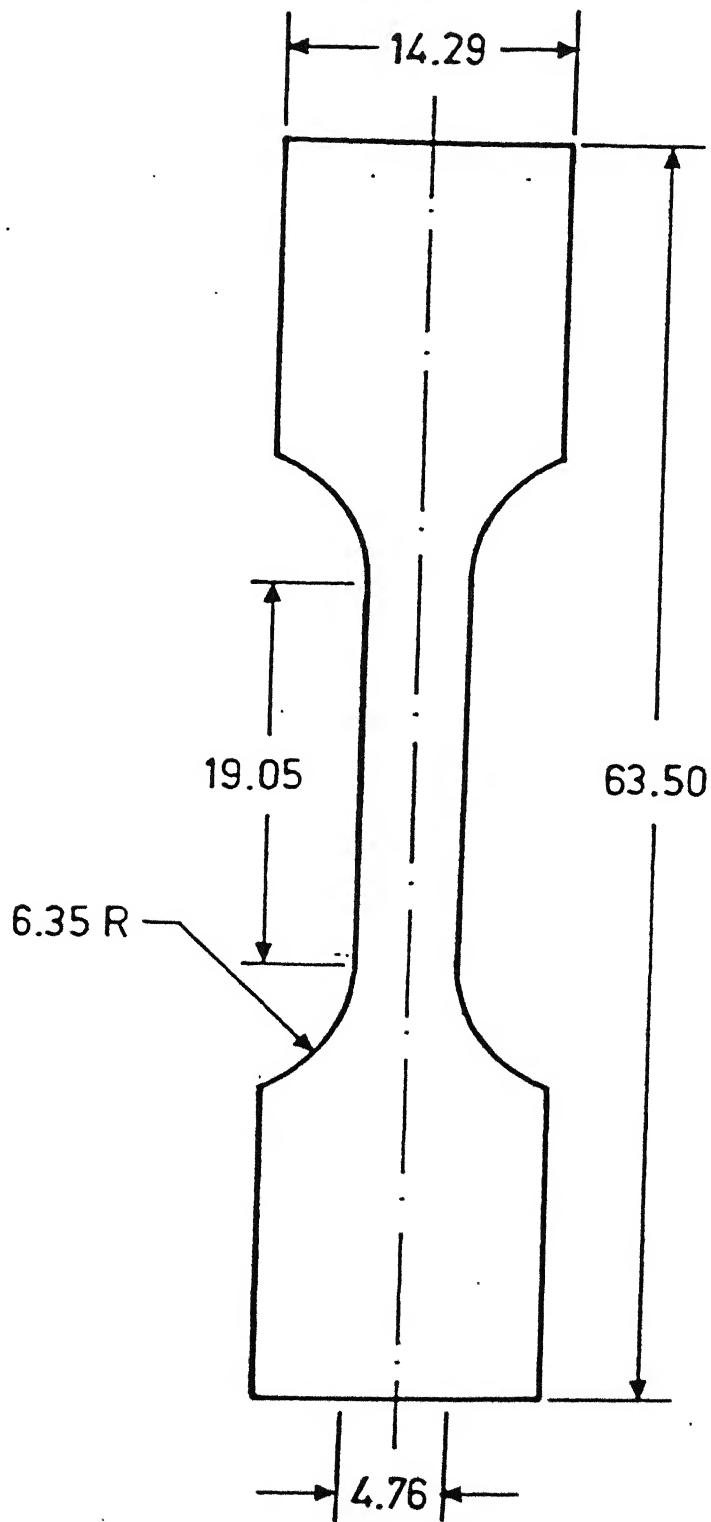


Fig.3.5 Tensile test specimen (units in mm)

3.4.3 METALLOGRAPHY :

The structure of strip at various stages of processing was examined under optical, scanning and Transmission Electron microscopes. Each of the above were discussed separately below.

3.4.3.1 OPTICAL MICROSCOPY :

Samples for optical microscopy were mounted in epoxy resin/plastic prior to their polishing. The polishing method consists of the following steps:

- 1) Samples were carefully ground on 1/0; 2/0; 3/0 and 4/0 emery papers using a light pressure.
- 2) Final polishing was done on microcloth wheel using a 0.3 μm alumina water slurry.

After final polishing the specimens were washed dried and etched with FeCl_3 solution the concentration and constituents given by KEHL⁽²¹⁾. After etching they were rinsed with warm water and were dried under a warm air blast.

3.4.3.2 SCANNING ELECTRON MICROSCOPY :

Since copper is electrically conducting, anycoating was not needed for observation of samples in SEM. The Fractured surface at different stages of processing were observed in JEOL. SEM. Microstructures in cold rolled and hot rolled strips were observed in SEM.

3.4.3.3 TRANSMISSION ELECTRON MICROSCOPY :

TEM samples were prepared from the hot rolled copper strips subjected to various treatments, by the following procedure:

- (1) The thin sample for electrojet polishing was prepared by mechanically thinning a piece of the copper strip to 0.05 mm.
- (2) The thin specimen obtained in (1) was used for the preparation of 3 mm dia disc samples for electrojet polishing. This was done by using a small punching equipment.
- (3) This 3 mm dia disc were further thinned by electrojet polishing until a perforation occur.
- (4) The region around the perforation will be very thin and this used for observation of structure.

The following were the conditions used for electrolytic jet polishing⁽²⁰⁾ :

Voltage → 15 V

Temperature → - 25°C

Electrolyte → Nitric acid and methanol in the ratio 1 : 2

The electrojet polishing procedure was described in⁽²³⁾. Samples after electrolytic jet polishing were washed in methanol twice and dried using a warm air blast.

The samples were stored in a capsule and kept under vacuum.

The samples were examined in JEOL Transmission Electron Microscope for microstructures.

3.4.4 GRAIN SIZE MEASUREMENTS :

The grain size; Aspect ratio were measured according to the Intercept method after Heyn⁽²⁴⁾.

3.4.5 QUANTITATIVE ANALYSIS OF INCLUSIONS:

The following quantitative parameters related to the inclusions present in atomized copper strip were determined:

- (1) Total number of inclusions.
- (2) Size distribution of inclusions.

(a) An Image analyser was used for determining the above parameters from hot rolled and annealed, and unetched samples.

(b) Quantitative analysis inclusion was done on samples cold rolled to different deformations using optical microscopy and following parameters were determined.

- (a) Total no. of inclusions and
- (b) No.of particles fragmented/decohesioned.

CHAPTER - 4

RESULTS AND DISCUSSIONS

4.1 EFFECT OF HOT ROLLING TEMPERATURE AND THICKNESS REDUCTION ON THE DENSIFICATION OF POROUS COPPER STRIP

The densification behaviour has been studied by hot rolling porous copper strip to various thickness deformation at two different temperatures, 1023K & 1223K, by measuring the relative density of strip corresponding to the deformation. The thickness reduction was measured by considering initial and final thicknesses.

The effect of hot rolling on the relative density of copper strip is shown in Fig. 4.1. Table 4.1(a) and 4.1(b) show the values of total porosity (ϵ_t), inter connected porosity (ϵ_{inter}) and isolated porosity (ϵ_{iso}) in the strip as a function of hot rolling deformation at 1023K and 1223K. The behaviour is graphically represented in Fig. 4.2 & 4.3.

Hot rolling behaviour of porous metal strips has recently been analysed by the plasticity theory of porous metals⁽²⁵⁾. The following analytical relationship has been obtained

$$\epsilon_t = 1 - \left[\left(\frac{\rho_0}{\rho} \right)^{\frac{C+3}{3}} \left(\frac{1 - \rho^2}{1 - \rho_0^2} \right)^{C/6} \right] \quad \dots \quad 4.1$$

where C is a constant having a value of 0.4, obtained from the

Table 4.1

Effect of Thickness Reduction on Densification of Porous
Copper Strip.

(a) Hot Rolling Temperature = 1023K

Relative Density of the starting strip, $\rho_0 = 0.35$

Fractional Thickness Reduction	Relative Density of HR strip ρ	Total Porosity of HR strip ϵ_T	Interconnected Porosity of HR strip ϵ_{inter}	Isolated Porosity HR strip ϵ_c
0.02	0.391	61	56	5.0
0.173	0.410	59	54	5.0
0.290	0.420	58	53.1	4.9
0.440	0.567	44	40.0	4.0
0.530	0.65	35	32.0	3.0

(b) Hot rolling temperature = 1223K

Relative density of the starting strip, $\rho_0 = 0.35$

Fractional Thickness Reduction	Relative Density of HR strip ρ	Total Porosity of HR strip ϵ_T	Interconnected Porosity of HR strip ϵ_{inter}	Isolated Porosity HR strip ϵ_c
0.05	0.395	61	56	5
0.19	0.420	58	52	6
0.27	0.440	56	51	5
0.35	0.459	54	50	4
0.49	0.600	40	36	4
0.63	0.800	20	17	3

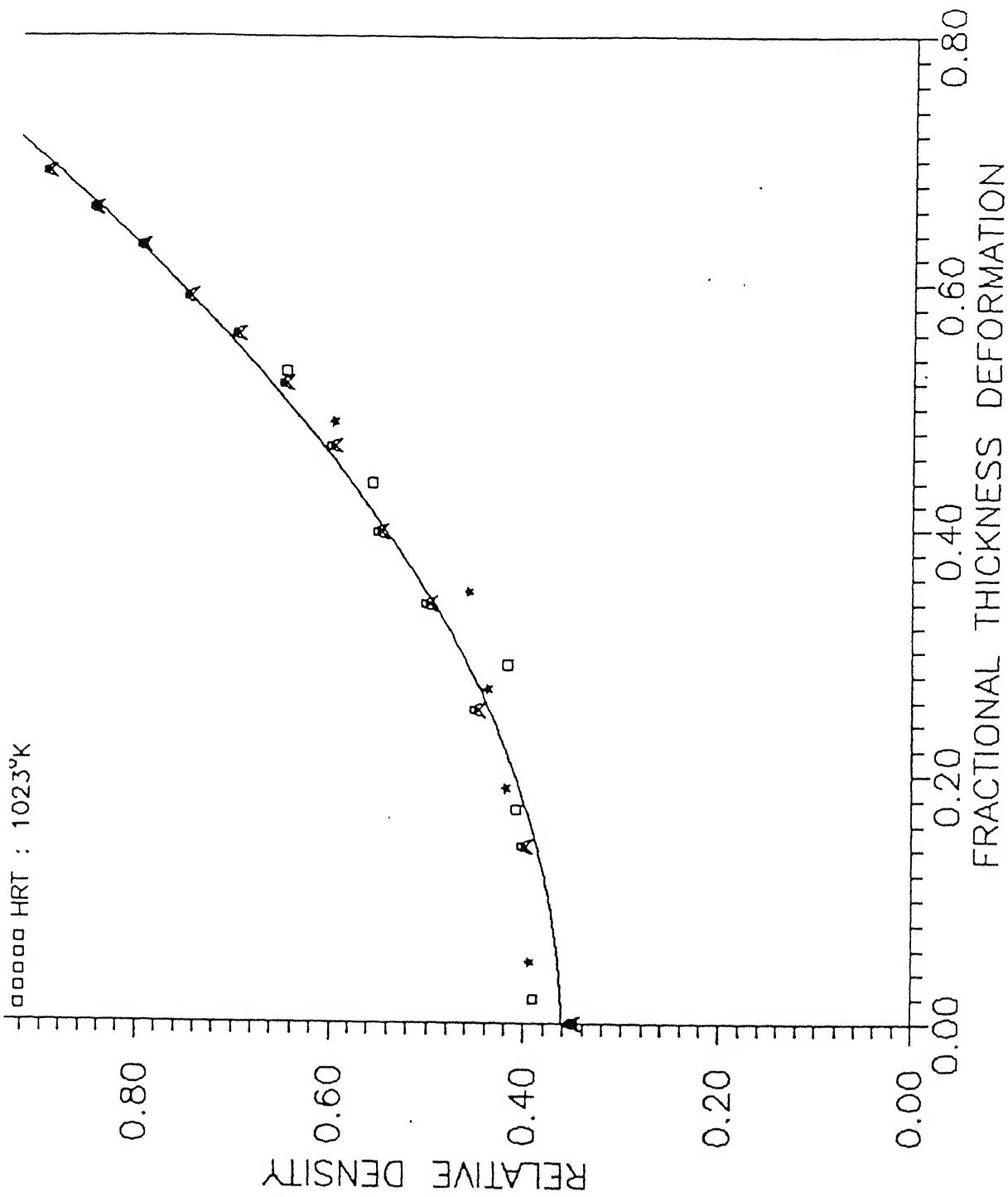
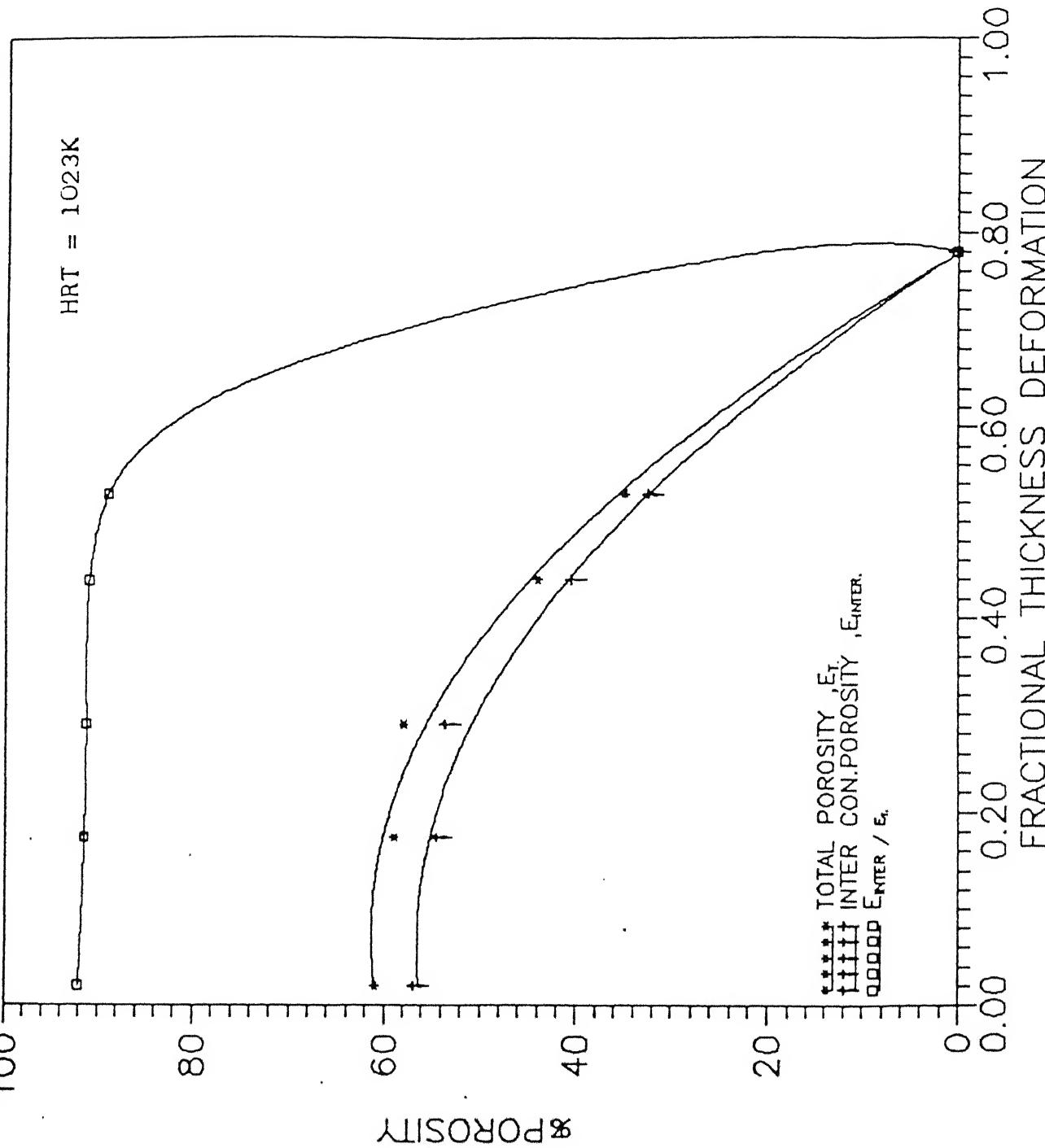


FIG 4.1 EFFECT OF HOT ROLLING TEMPERATURE AND THICKNESS REDUCTION ON STRIP DENSIFICATION



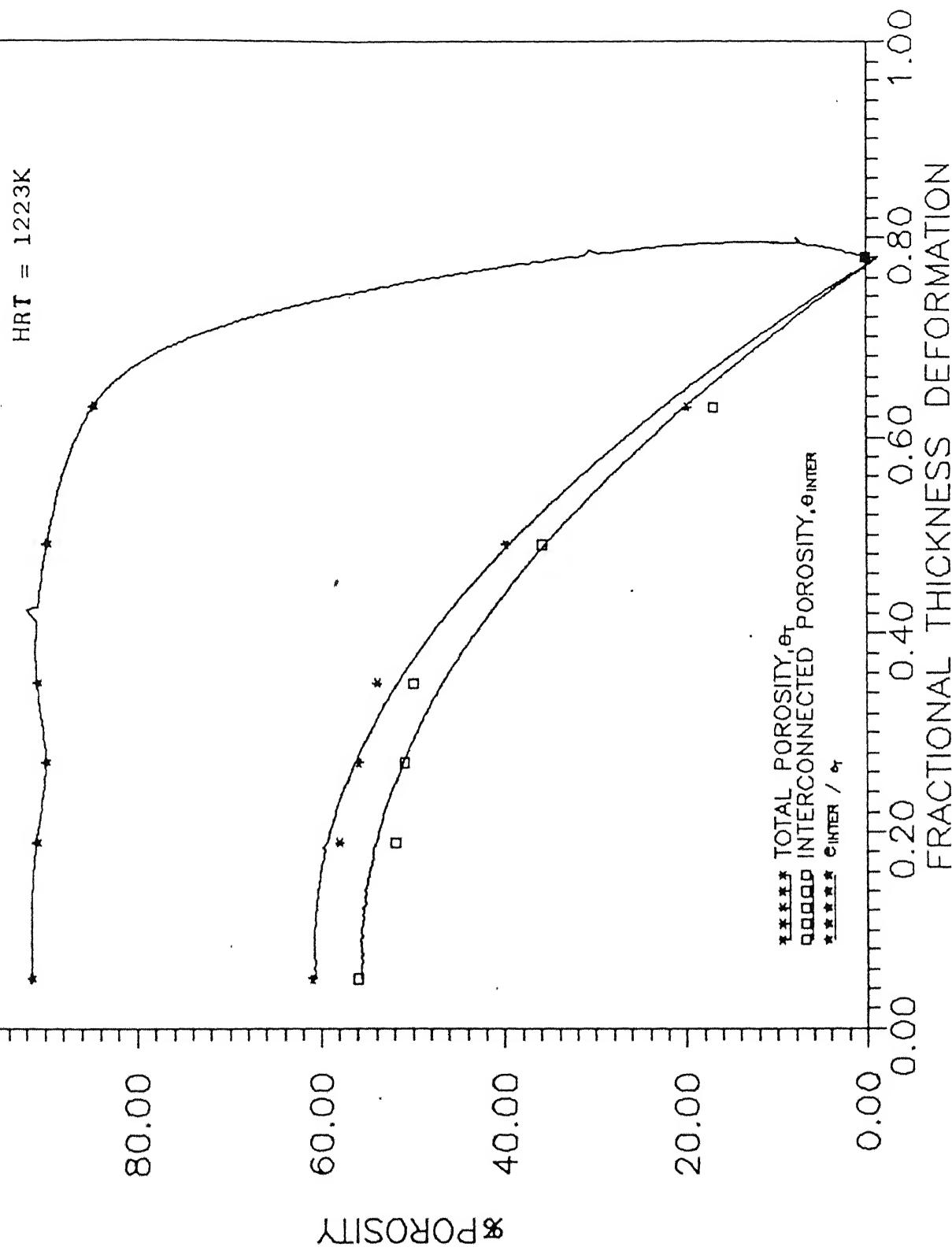


FIG. 4.3. EFFECT OF THICKNESS REDUCTION ON POROSITY

ratio of youngs modulus to shear modulus of copper.

e_t is the fractional thickness deformation

ρ_0 ; ρ are the relative densities of strip before and after deformation.

The above equation predict the temperature independency of densification. The experimental values for two hot rolling temperature were found to agree well with the theoretically predicted densification behaviour from Eqn. 4.1, as shown in Fig. 4.1.

Densification, in porous strips, due to hot rolling can be considered to be occurring through the following mechanisms.

- a) Transitional restacking of particles
- b) Contact growth
- c) Longitudinal flow of the particles.

(a) The starting porous strip consisted of basically an aggregate of loosely sintered copper particles. The overall stresses acting on the strip during initial stages of deformation, compressive in thickness direction and Tensile in the rolling direction, are too small to cause any plastic deformation of the particles since no substantial plastic deformation of the bulk is possible during initial stages of deformation i.e. low thickness reductions. Particles after their sliding tend to restack themselves in void regions available in their vicinity where they can be accomodated easily without any major plastic deformation. Such a transitional restacking of particles with little plastic flow is shown

schematically in Fig. 4.4 (a) Fig. 4.5 show the SEM views of strip hot rolled at 1223K.

It has been proposed⁽²⁶⁾ that random dense packing can be attained at a density of 0.67 of theoretical density. The deformation range corresponding to this density is 0.53 - 0.6. From SEM views it can be inferred that transitional restacking of particles is the predominant mechanism upto a Fractional thickness deformation of 0.5. During transitional restacking, most of the strain given will be used for rearrangement and less is consumed for plastically deforming the particles.

(b) The increase in Fractional thickness deformation above 0.6 causes the particles to come much closer thus decreasing the interconnected porosity. Particles from the random dense packed stage come closer to each other thus increasing the interparticle contact area, as the thickness deformation increases. Since most of the interconnected porosity is eliminated by the close packing of particles, only isolated porosity remains. Flattening of particles also occurs to some extent in this case.

(c) The Longitudinal flow of the particles is predominant at higher thickness reductions, since the strain given to the strip is utilized to maximum extent to plastically deform the particles as there is little amount of porosity. As thickness deformation further increases giving the relative density value near to theoretical, the porosity decrease to minimum.

Since the volume changes at this stage are small, the

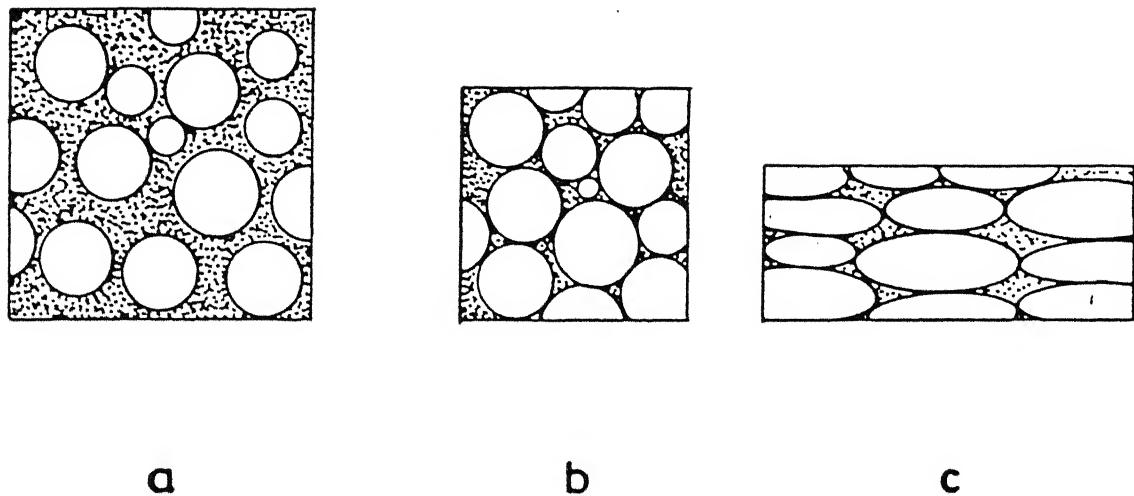


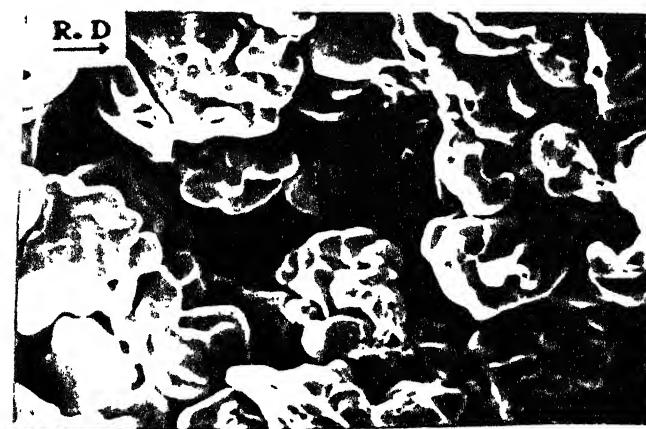
Fig.4.4 Schematic view showing three stages of densification of porous copper strip during its hot rolling



a 17.5% HR (X1500)



b 29% HR (X1500)





d 53% HR (X1500)



e 61% HR (X1000)



f 61% HR (X2000)

Fig. 4.5 SEM view showing particle arrangement at different stages of densification of porous copper strip during its hot rolling.



Fig 4.5.1 SEM view showing longitudinal flow in particles of a fully dense strip during its hot rolling. (X500)

thickness deformation given to the strip results in almost equivalent elongation in rolling direction.

4.2 EFFECT OF HOT ROLLING TEMPERATURE ON STRUCTURE AND MECHANICAL PROPERTIES OF FULLY DENSIFIED COPPER STRIPS

Results on the densification behaviour of porous metal strips as discussed in section 4.1 showed that the densification of the strip is unaffected by hot rolling temperature. However, the evolution of microstructure developed during the densification rolling of porous copper strip is likely to be affected by hot rolling temperature. In order to study the development of microstructure in the strip, different sets of experiments were carried out. In the first set of experiments fully densified strips hot rolled at different temperatures were quenched in water within 5 sec of their rolling. This procedure ensured the retention of as rolled microstructure in the strip without causing any static recovery or recrystallisation taking place during their cooling. In the second set of experiments the strips were cooled under graphite chips so that static softening processes, if any, can occur.

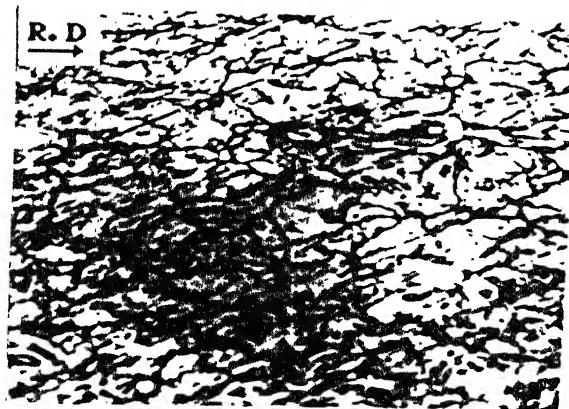
4.2.1 MICROSTRUCTURES OF FULLY DENSIFIED STRIPS

Optical, scanning as well as Transmision electron microscopy methods were used to examine the microstructure of various hot rolled strips.

Fig. 4.6 and 4.7 show the optical micrographs of the



a 923K (X200)



b 1023K (X200)

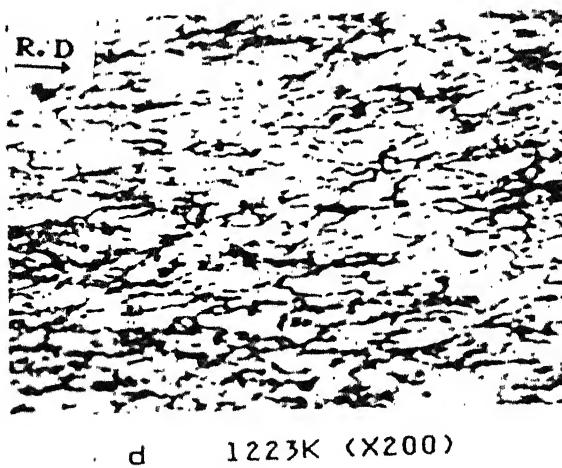
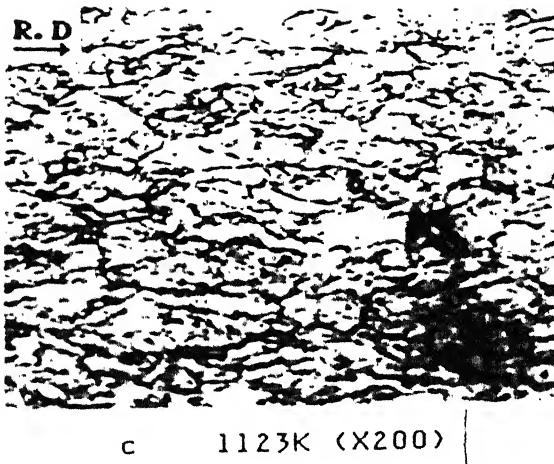
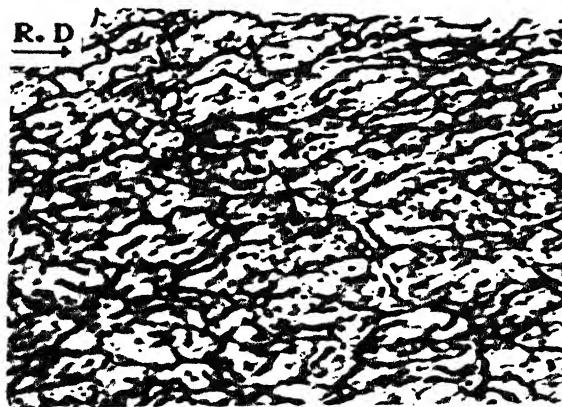


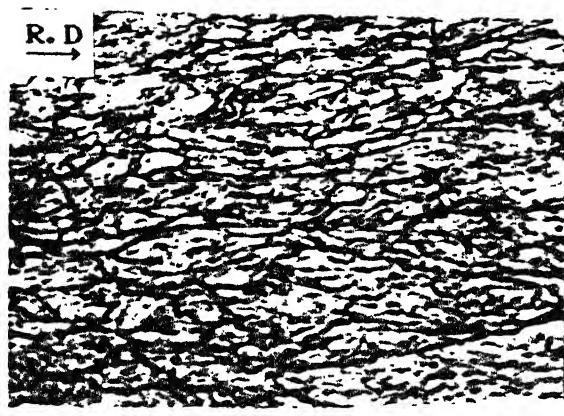
Fig. 4.6 Optical micrographs in thickness direction, of strips hot rolled at different temperatures and quenched.



a 923K (X200)



b 1023K (X200)



C 1123K (X200)



d 1223K (X200)

Fig. 4.7 Optical micrographs in thickness direction of strips hot rolled at different temperatures and cooled in graphite chips.

thickness cross section parallel to rolling direction, of strips rolled at 923K, 1023K, 1123K and 1223K for the two sets of strips. It can be seen that all these microstructures do not show any porosity which was commensurate with density measurements done on these strips. However, all the microstructures showed elongated grains and aspect ratio of grains was found to be different in strips rolled at different temperatures. Further at 923K the grains were of mixed size.

Fine inclusions presumably that of Cu_2O were found to be present in microstructures. The size distribution of inclusions present in strips hot rolled at 1223K was obtained by using Image Analyser by examining the microstructure under unetched condition. The inclusion size distribution histogram is shown in Fig. 4.8. It is clear from the histogram that the size distribution of inclusions in the strip was positively skewed i.e. most of the inclusions were in the micron and submicron size range with relatively few inclusions present in maximum size range of 10-14 μm .

Aspect ratio of grains obtained in strips rolled at different temperatures was calculated by measuring grain size in transverse as well as in Longitudinal directions. Aspect ratio of grains as a function of hot rolling temperature is shown graphically in Fig. 4.9 for two sets of strips.

It can be seen from the above figure that aspect ratio decreases as the hot rolling temperature increases. This can be explained by considering that at high hot rolling temperatures

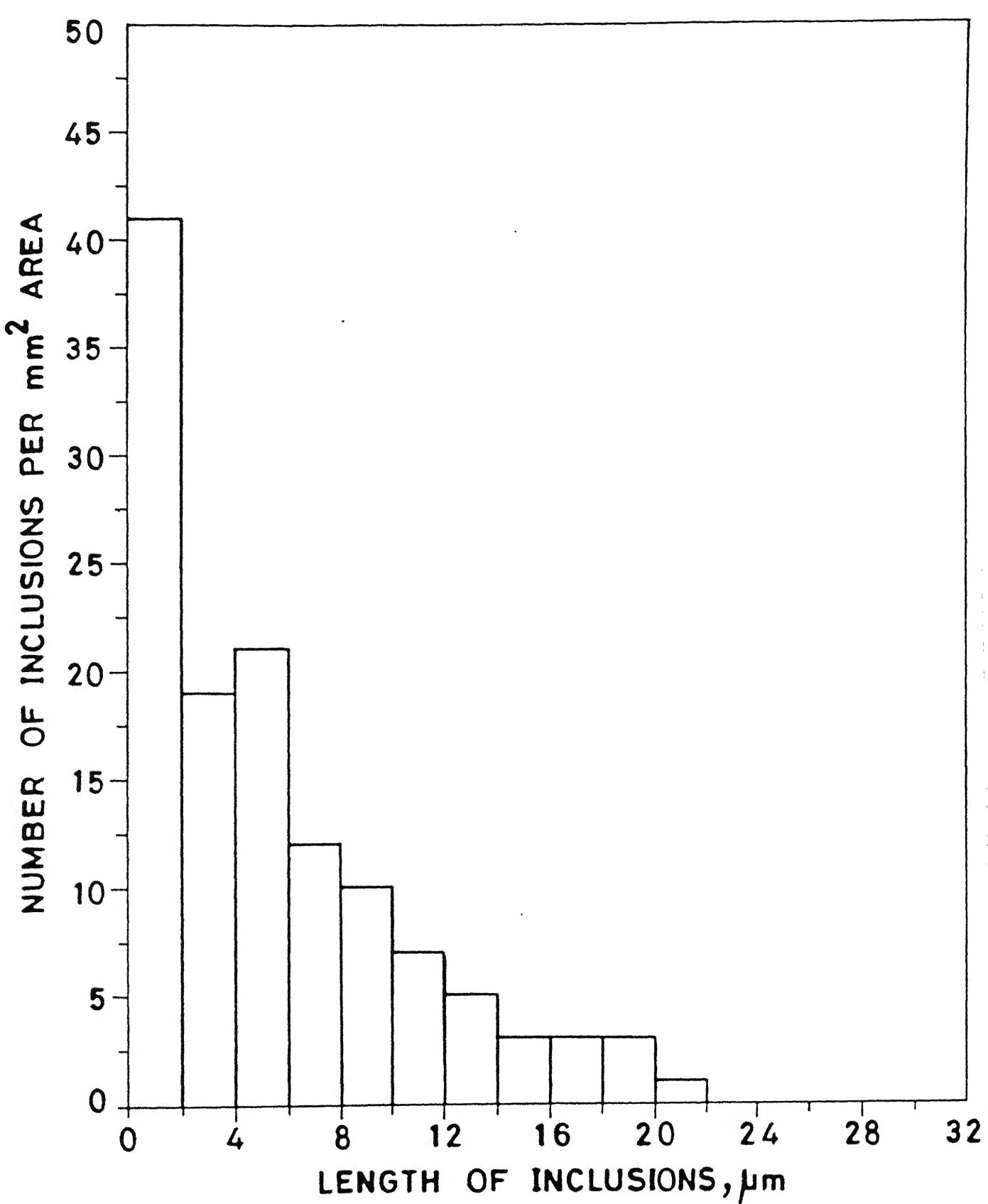


Fig.4.8 Inclusion size distribution histogram for copper strip prepared from atomized copper powder by hot rolling at 1223K & annealed at 823K

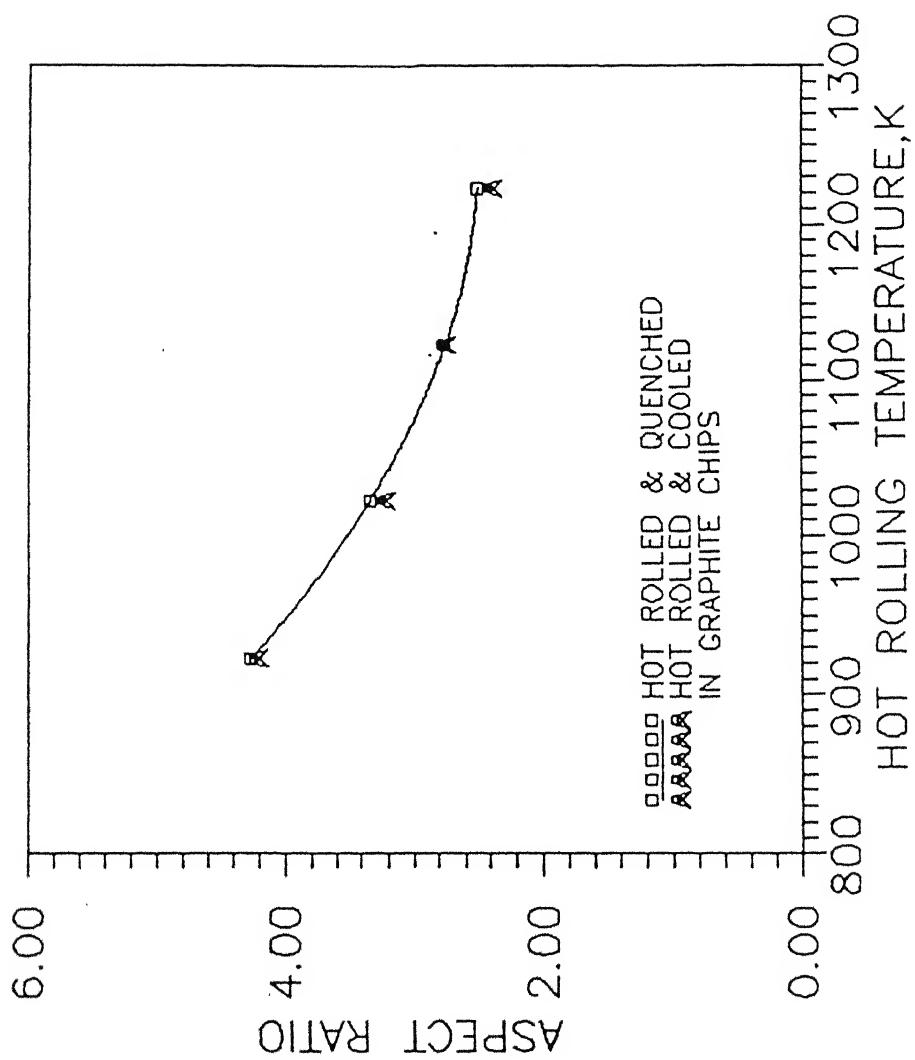
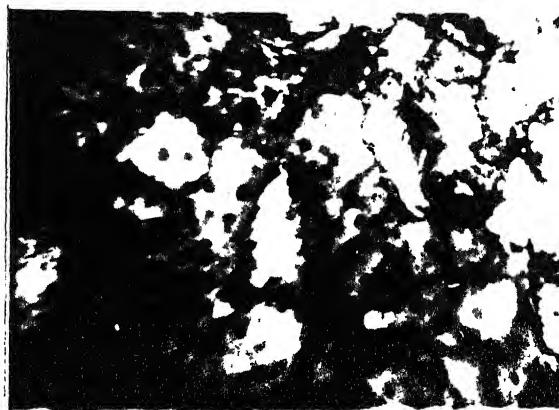


FIG 4.9 EFFECT OF HRT ON ASPECT RATIO OF GRAINS

thermal energy assistance is more compared to that low hot rolling temperatures. Dynamic softening can take place at high hot rolling temperatures giving a low aspect ratio. For the second set of strips, hot rolled at different temperatures and cooled in graphite chips, the aspect ratios are similar compared to above at corresponding temperatures.

Transmission electron micrographs of the strips rolled at 923K, 1023K, 1123K and 1223K are shown in Fig. 4.10 and 4.11. It can be seen that all these micrographs showed the dislocation cell structure. However, all the microstructures showed different cell wall thickness for strips hot rolled at different temperatures. The occurrence of dynamic softening mechanisms is dependent on many factors such as strain, temperature of deformation and stacking fault energy of the metal. According to JONAS et al⁽²⁷⁾. In processes where strain is low the grains soften by dynamic recovery and in metals of low stacking fault energy, static recrystallization, if at all permitted, takes place after dynamic recovery. In case of copper strip made from hot rolling route the actual strain in the strip will be imparted to the copper particles present much less than that calculated from the thickness reduction values, because most of the strain is used for filling of pores by restacking of particles. Also the stacking fault energy of copper is low. Therefore dynamic recovery, if any, can occur rather than dynamic recrystallisation. This is the reason for the presence of dislocation cell structure. For the second set of strips the static recrystallisation has not occurred to a greater extent thus giving the cell structure.



a 923K (X30,000)



b 923K (X60,000)



c 122K (X30,000)

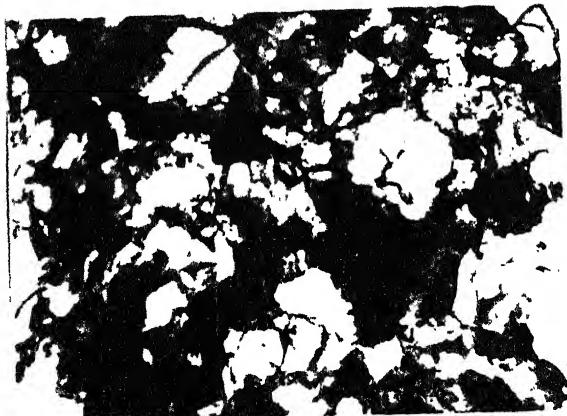
Fig. 4.10 Transmission electron micrographs showing the dislocation cell structure for fully dense hot rolled strips and quenched.



a 923K (X30,000)



b 923K (X1,20,000)



c 1223K (X20,000)

Fig. 4.11 Transmission electron micrographs showing the dislocation cell structure for fully dense hot rolled strips and quenched.

4.2.2 MECHANICAL PROPERTIES OF FULLY DENSIFIED STRIPS

The effect of hot rolling temperature on the mechanical properties of the strips rolled at 923K, 1023K, 1123K and 1223K are shown in Figs 4.12, 4.13 and Figs 4.14, 4.15 for two different sets of strips. Table 4.2 and 4.3 show the values of Tensile strength, 0.2% yield strength and elongation as a function of hot rolling temperature for two set of strips.

Fractography study was done on the fractured tensile samples of hot rolled strips with scanning electron microscope. Fig 4.16 and Fig 4.17 shows the fractographs of strips hot rolled at different temperatues for the two sets of strips. It can be seen that the fractured surface at high temperature hot rolling showed a particilly dimpled surface and for low hot rolling temperature an intergramular fracture was observed.

It can be seen from Fig. 4.12 to Fig. 4.15 the values of mechanical properties increased with increase in hot rolling temperature. The reasons for this nature are discussed below. The factors that can contribute the variation in mechanical properties of rolled copper strips are porosity, inclusions, dislocations and bond strength of particles. Optical micrographs showed no porosity and low content of inclusions so the predominant factors are dislocations and bond strength of particles. Transmission electron micrographs showed dislocation cell structure in both cases which implies that a high density of dislocations is present in the strips. This is the reason for the

Table 4.2

EFFECT OF HOT ROLLING TEMPERATURE ON MECHANICAL PROPERTIES
OF STRIPS HOT ROLLED AND QUENCHED

Hot Rolling Temperature, K	U.T.S. MN/m ²	0.2% Yield Strength MN/m ²	% Elongation
1223	328; 332	240	5.98
1123	339; 327	228; 216	3.05
1023	302; 279	196; 209	1.50
923	248; 247	187.4; 187	1.11

Table 4.3

EFFECT OF HOT ROLLING TEMPERATURE ON MECHANICAL PROPERTIES
OF STRIPS HOT ROLLED AND COOLED IN GRAPHITE CHIPS

Hot Rolling Temperature, K	U.T.S. MN/m ²	0.2% Yield Strength (MN/m ²)	% Elongation
1223	335	243	6.1
1123	336, 338	227.2	3.71
1023	312	212	1.68
923	251	191	1.1

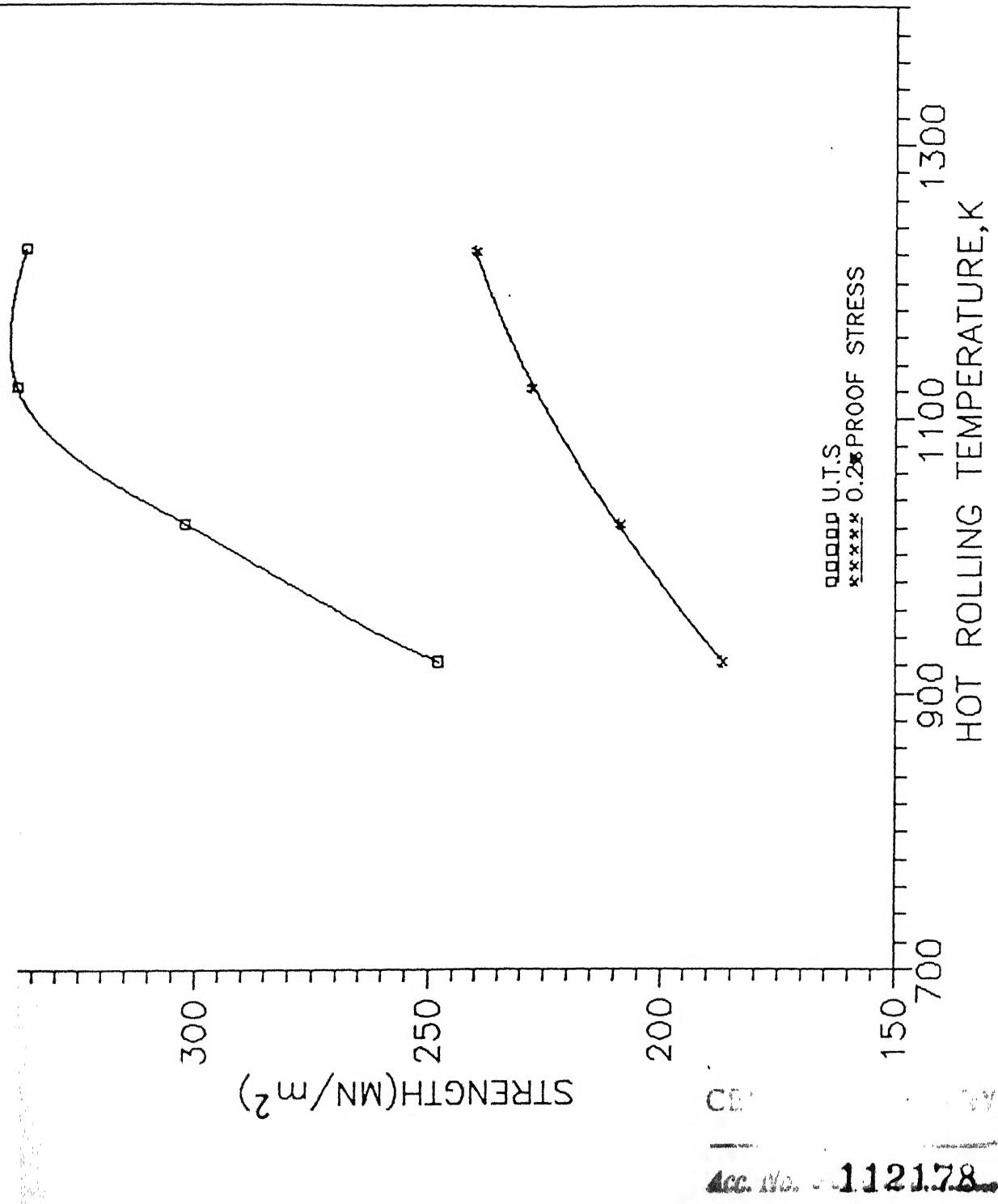


FIG 4.12 .EFFECT OF H.R.T. ON U.T.S. & 0.2% PROOF STRESS
OF A STRIP HOT ROLLED TO FULL DENSITY & QUENCHED

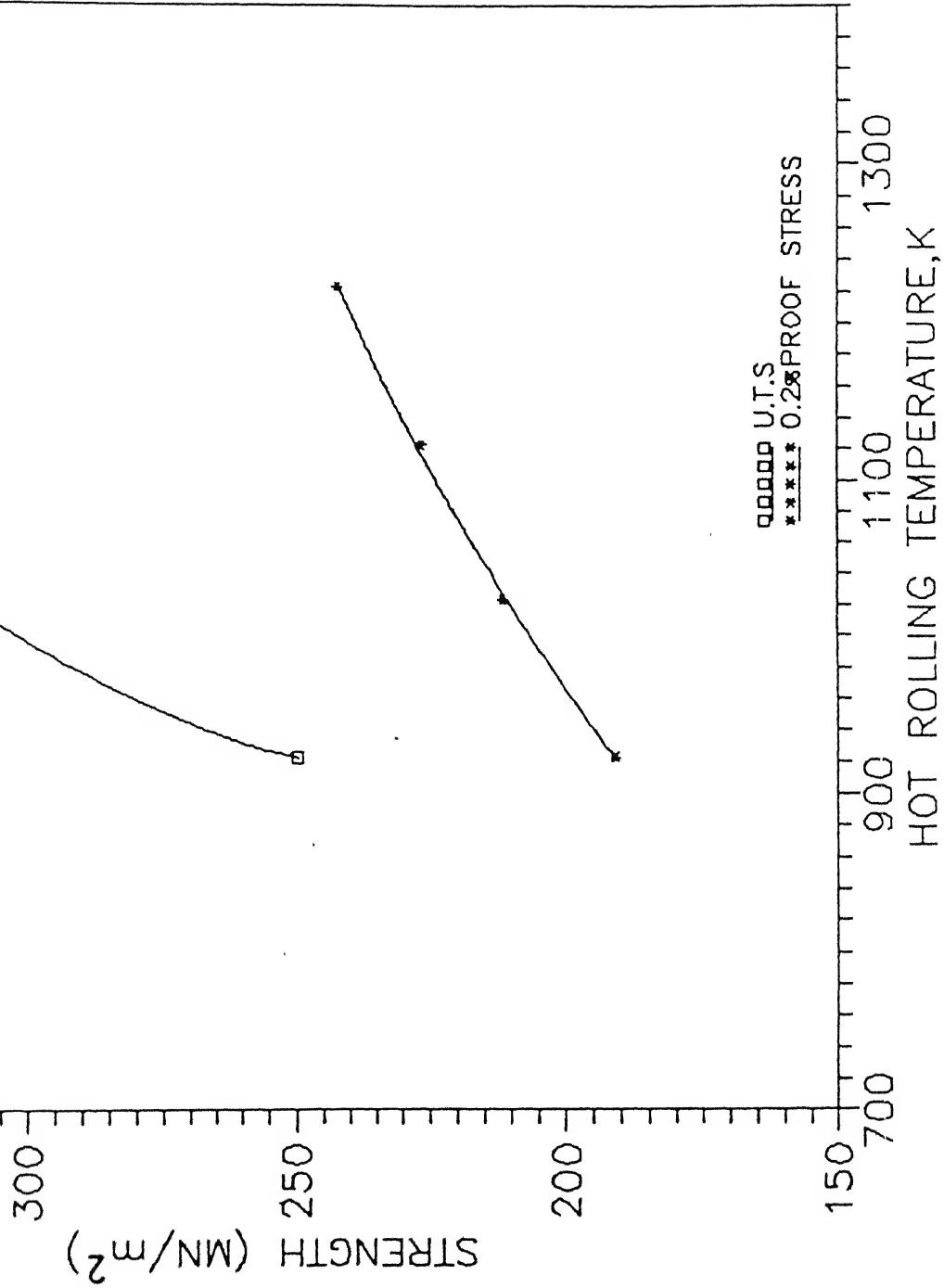


FIG 4.13 .EFFECT OF H.R.T. ON U.T.S. & 0.2%PROOF STRESS
OF A STRIP HOT ROLLED TO FULL DENSITY &
COOLED IN GRAPHITE CHIPS

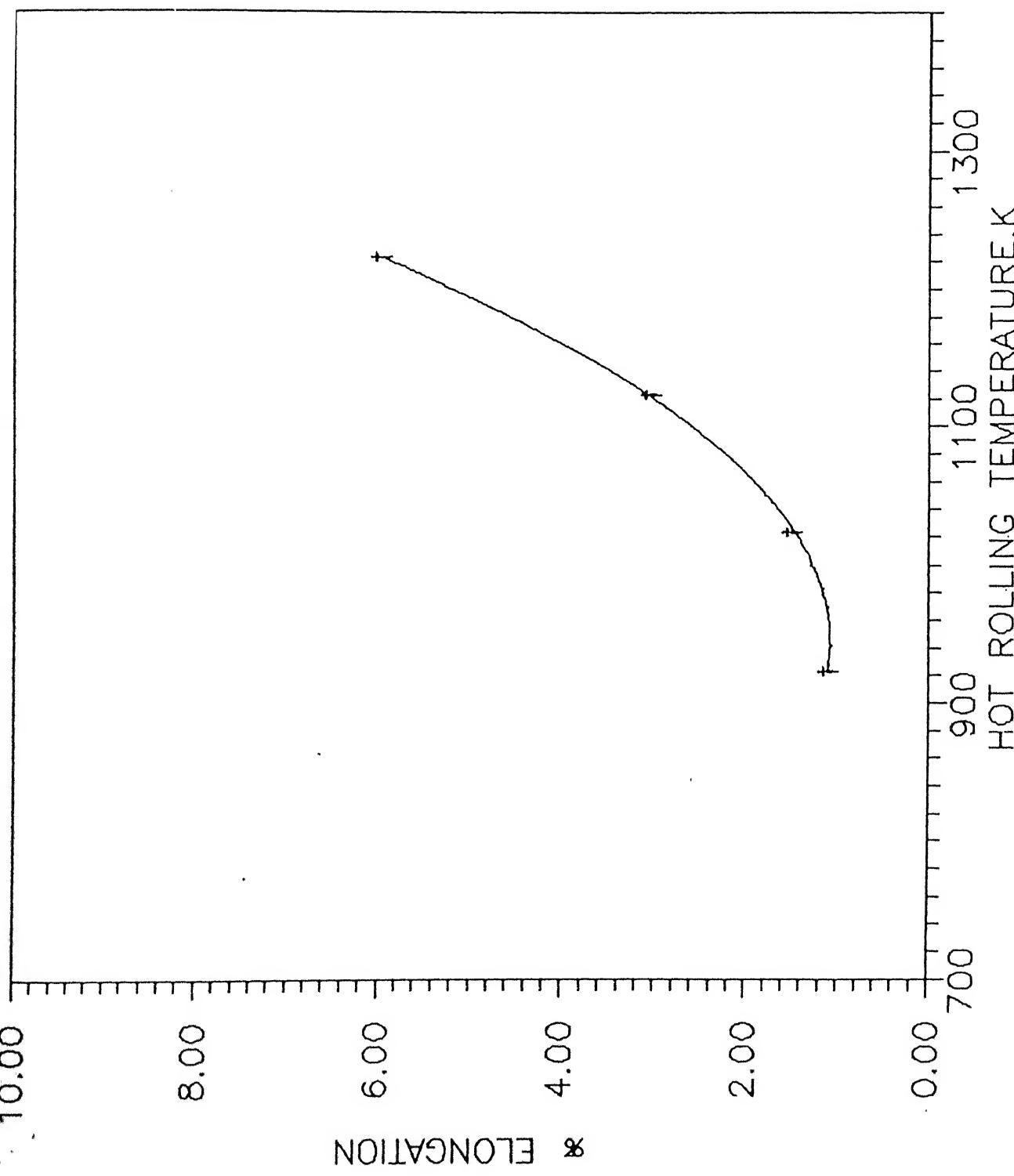


FIG. 4.14. EFFECT OF HRT ON ELONGATION OF STRIP
DUE TO PLASTICITY. Δ INFLUENCE

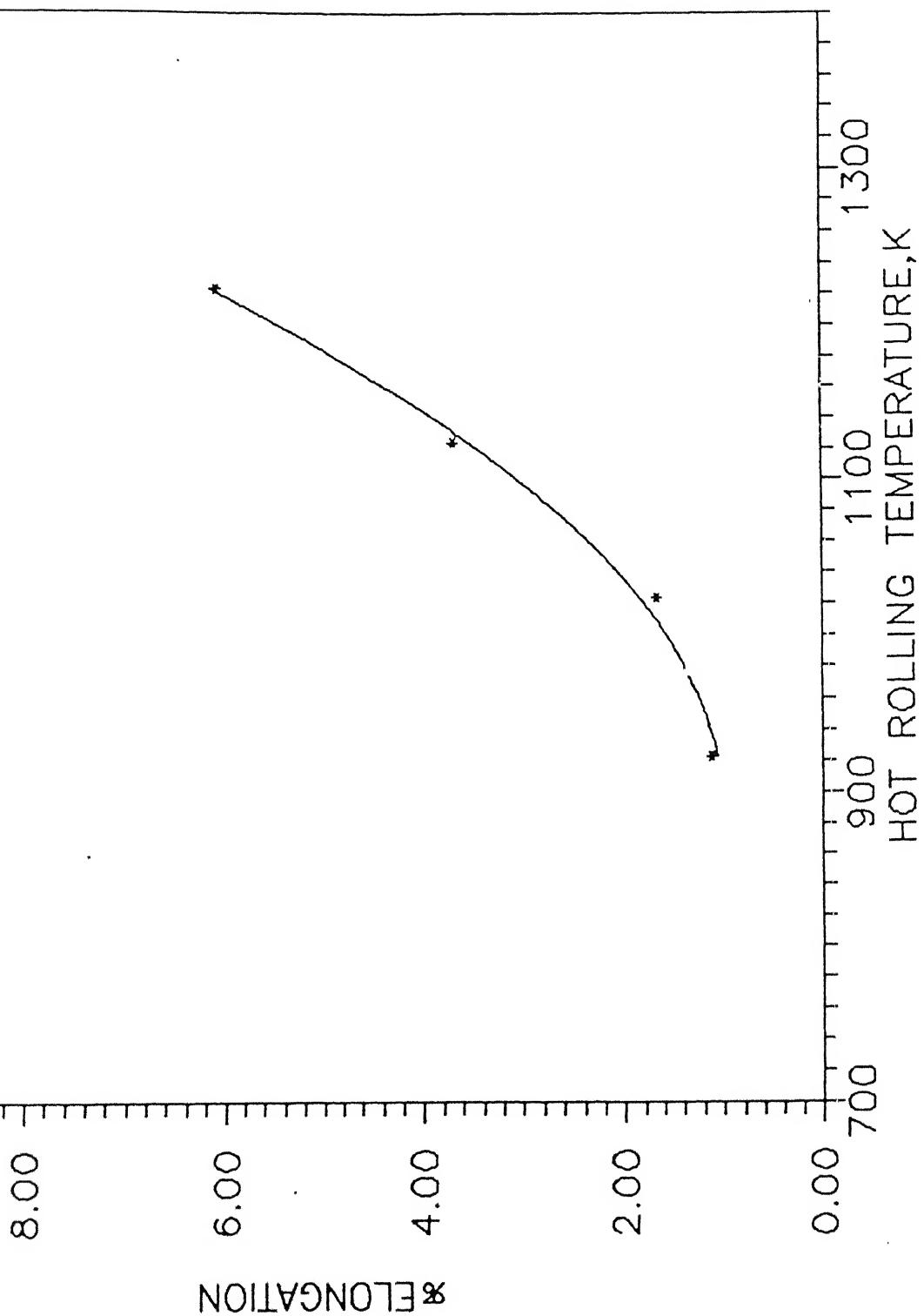


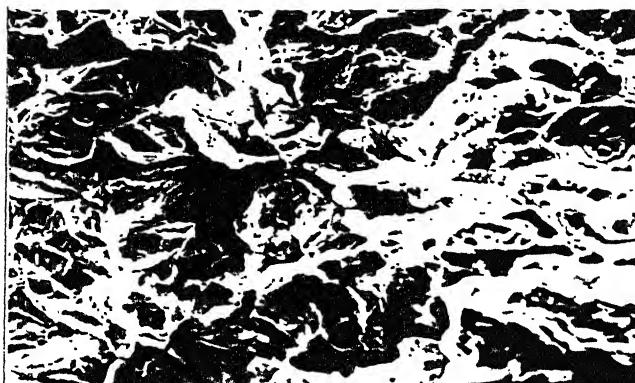
FIG 4.15 .EFFECT OF HRT ON ELONGATION OF A STRIP HOT ROLLED TO FULL DENSITY & COOLED IN GRAPHITE CHIPS



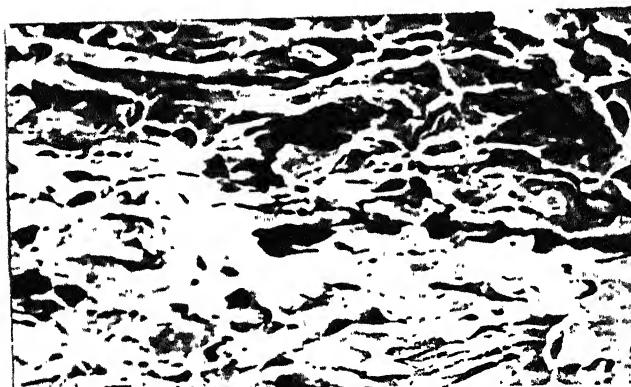
a 923K (X1000)



b 1023K (X1000)



c 1123K (X1000)

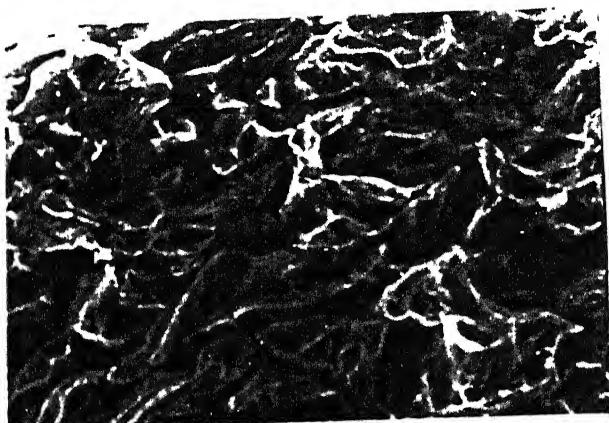


d 1223K (X1200)

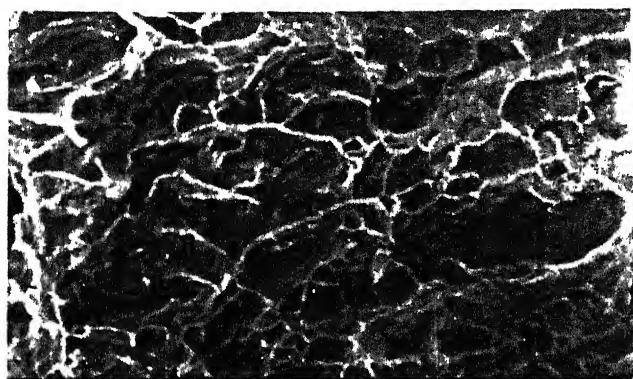
Fig. 4.16 SEM view of fractured surfaces after the tensile test on strips hot rolled at different temperatures and quenched.



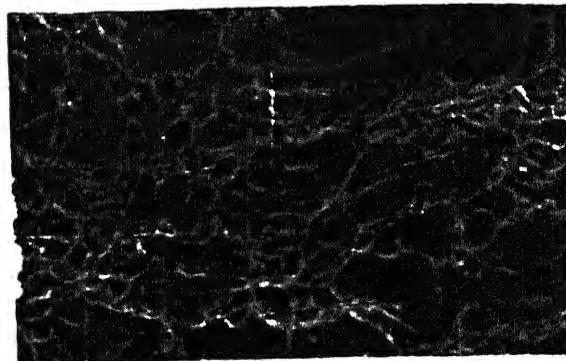
a 923K (X1200)



b 1023K (X1500)



c 1123K (X1500)



d 1223K (X2300)

Fig. 4.17 SEM view of fractured surfaces after the tensile test on strips hot rolled at different temperatures and cooled in graphite chips.

high strength and low elongation values of hot rolled strips.

The tensile strength and elongation values increased as the hot rolling temperature increases. This is due to the good bond strength of particles at high hot rolling temperatures as revealed by scanning electron micrographs of Fractured surfaces. The same nature was observed with second set of strips TEM micrographs of this above hot rolled strips also showed the dislocation cell structure and the mechanical properties were similar thus showing that static no softening mechanisms have not taken place to a greater extent in the second set. The increase in 0.25 yield strength with increase in hot rolling temperature might be due to easy plasticity occurring at low hot rolling temperatures where the bond strength is low. For the second set of strips hot rolled and cooled in graphite chips, the same nature was observed.

4.3 EFFECT OF ANNEALING ON STRUCTURAL AND MECHANICAL PROPERTIES OF FULLY DENSIFIED COPPER STRIP

A set of sintered copper strips were hot rolled at 1223K to full density in a single pass. These were subsequently annealed at 823K for 1800 sec in Argon atmosphere.

4.3.1 MICROSTRUCTURES OF FULLY DENSE ANNEALED STRIPS

Optical, scanning as well as Transmission electron microscopy methods were used to examine the microstructure of hot rolled and annealed strip. Fig. 4.18 shows the optical micrograph

of the copper stgrip hot rolled at 1223K and followed by annealing, at the thickness cross section parallel to rolling direction. It can be seen that the microstructure consisted of recrystallised grains.

Scanning electron micrograph of Fractured surface of Tension test sample is shown in Fig. 4.19. The structure showed a dimpled surface thus revealing the ductile fracture of the sample.

Transmission electron micrographs of the strip are shown in Fig. 4.20. It can be seen from Fig. that the dislocation density was less compared to that of as hot rolled sample in which no annealing was carried out. This is due to recrystallisation, taking place during annealing, by annihilating the dislocations present in the strip.

4.3.2 MECHANICAL PROPERTIES OF FULLY DENSE ANNEALED STRIPS

The mechanical properties of fully dense strip which was obtained by hot rolling the porous copper strip at 1223K to 82% thickness reduction are listed below:

Tensile strength = 202 MN/m²

Elongation = 40%

0.2% yield strength = 80 MN/m²

The increase in ductility and decrease in strength in the annealed strips as compared to the as hot rolled strips is due to the annihilation of dislocations during annealing.



Fig. 4.18 Microstructure of copper strip hot rolled at 1223K and annealed at 823K. (x200)

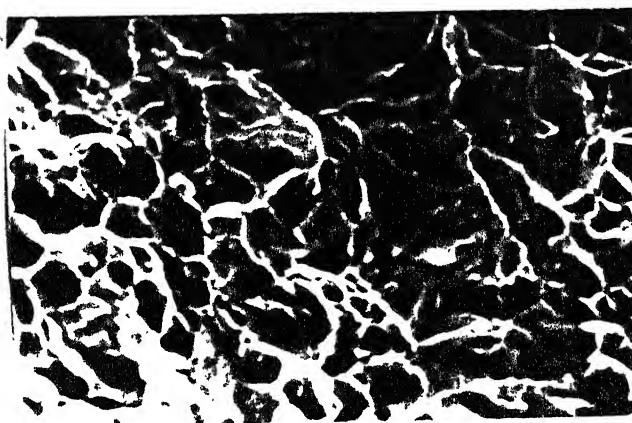


Fig. 4.19 SEM view of fractured surface after the tensile test on strip hot rolled at 1223K and annealed at 823K. (x3000)



Fig. 4.20 TEM view of strip hot rolled at 1223K and annealed at 823K. (x1,00,000)

4.4 EFFECT OF COLD ROLLING THICKNESS REDUCTION ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF FULLY DENSE HOT ROLLED COPPER STRIPS.

A set of copper strips were hot rolled at 1223K to full density in two passes and were subsequently cooled under graphite chips. A total of 82% thickness reduction was given. The fully dense hot rolled strips thus obtained were subsequently annealed in Argon atmosphere at 823K for 1800 sec. Annealing in hydrogen at higher temperatures for copper strips containing Cu_2O inclusions causes embrittlement⁽²⁸⁾. After the annealing treatment these strips could be further cold rolled upto 75% thickness deformation without any intermediate annealing. Tensile samples were prepared from the strips cold rolled to 10, 20, 40, 50 and 62 percent thickness reduction which were subsequently annealed at 823K in Argon atmosphere. The values of mechanical properties are listed in Table 4.4.

The hot rolled copper strips contained Cu_2O inclusions and the quantitative data regarding their size distribution are presented in Fig. 4.8. It was reported by earlier workers⁽²⁹⁻³⁷⁾ that during cold working of metals containing non metallic inclusions there can be decohesion and/or Fragmentation of inclusions due to the difference in plasticity between matrix and inclusion.

A common method to study the decohesion and/or Fragmentation of inclusions as a result of cold working is to observe thin films of material under electron microscope.

Table 4.4

EFFECT OF COLD ROLLING THICKNESS REDUCTION ON MECHANICAL
PROPERTIES OF COPPER STRIPS

Thickness reduction by cold rolling, %	Elongation, %	UTS MN/m ²
0	40.2	202
10	40	208
19	40	212
40	49.0	226
62	35.0	190

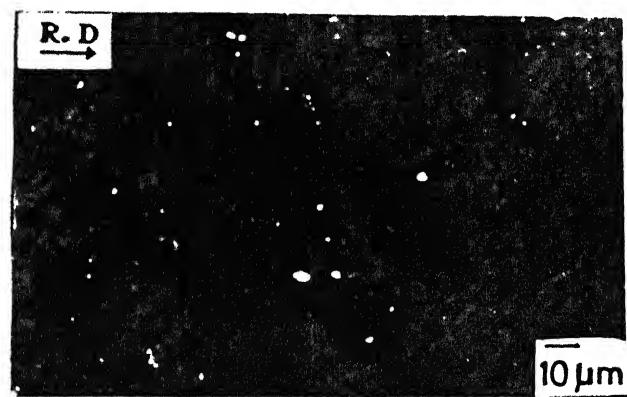
Table 4.5

EFFECT OF COLD ROLLING THICKNESS REDUCTION ON DECOHESION/
FRAGMENTATION OF INCLUSIONS

Thickness reduction %	Particles Fragmented/ Decohesioned, %
0	0
10	8
19	12
40	42
50	62



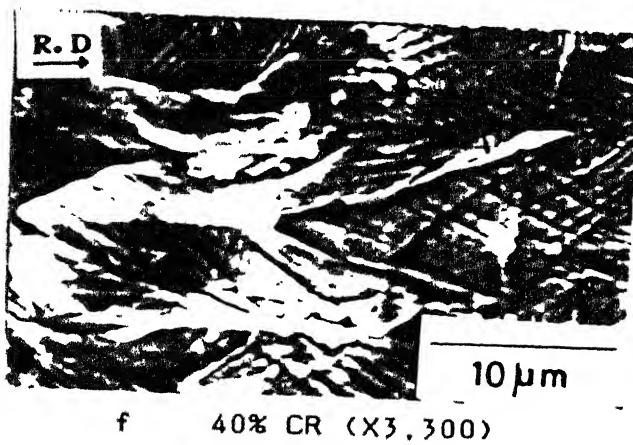
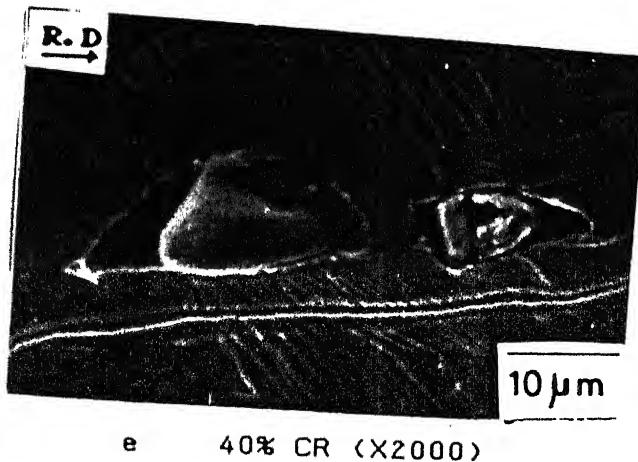
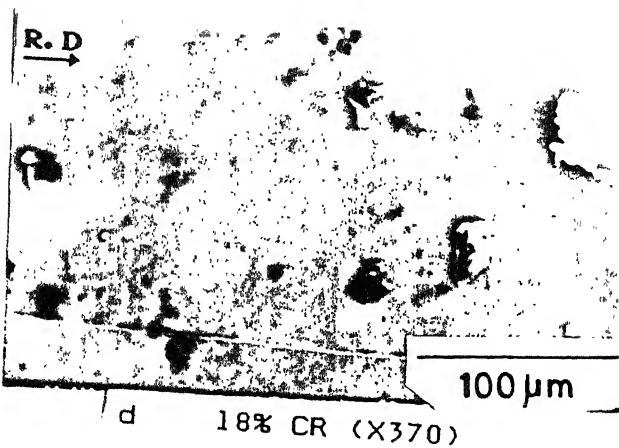
a 0% CR (X500)



b 0% CR (X550)

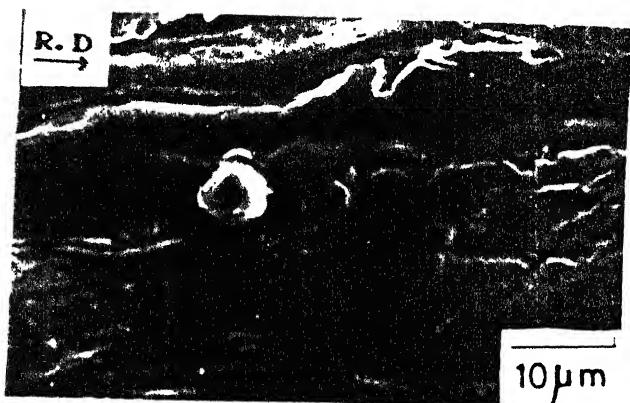


c 10% CR (X850)

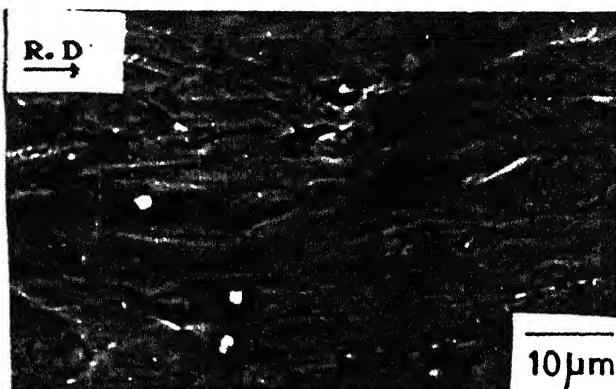




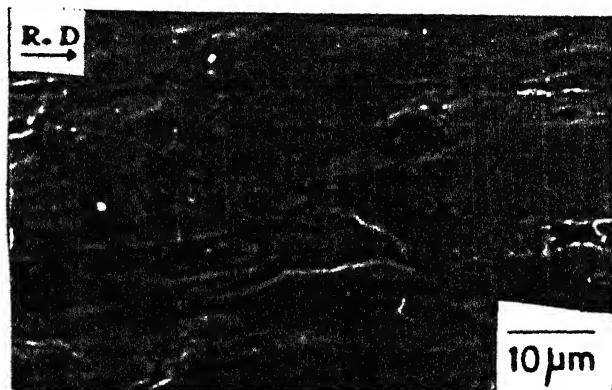
g 50% CR (X4,000)



h 50% CR (X1800)



i 62% CR (X1500)



j 62% CR (X1500)

Fig. 4.21 SEM views of fully dense copper strip, prepared from atomized copper powder cold rolled to different deformations, showing decohesion and/or fragmentation of Cu_2O inclusions.

cold rolled to a thickness deformation of 50%. But inclusions of sub micron sizes did not show any decohesion phenomenon even after cold rolling to 62% thickness reduction. Fig. 4.21 - show that submicron size inclusions have not undergone decohesion and/or Fragmentation in the copper strip cold rolled to 62% thickness deformation. Thus it can be observed that smaller the particles larger is the cold rolling thickness reduction for decohesion. The explanation for such a behaviour is given later.

The number of inclusions Fragmented and/or separated is calculated as a function of thickness reduction. Fig. 4.22 show the variation of decohesioned and/or Fragmented inclusions as a function of cold rolling thickness reduction. Optical microscope was used for counting the Fragmented and/or separated particles. Table 4.5 show the number of particles. Decohesioned/Fragmented with respet to cold rolling thickness reduction.

Copper strips can also be made directly from preformed cuprous oxide preforms⁽³⁸⁾. The method consists of reducing the cuprous oxide preform at K in H₂ atmosphere, followed by hot rolling to full density. The strip is further cold rolled and annealed. such copper strip contains about 1.2 vol % Cu₂O inclusions uniformly distributed in the copper matrix. The size distribution of inclusions is shown in Fig. 4.23. Such copper strip contains more Cu₂O inclusions than the copper strips used in the present investigation. Moreover, the average size of inclusion is also higher in the former strip.

In order to compare the decohesion/Fragmentation

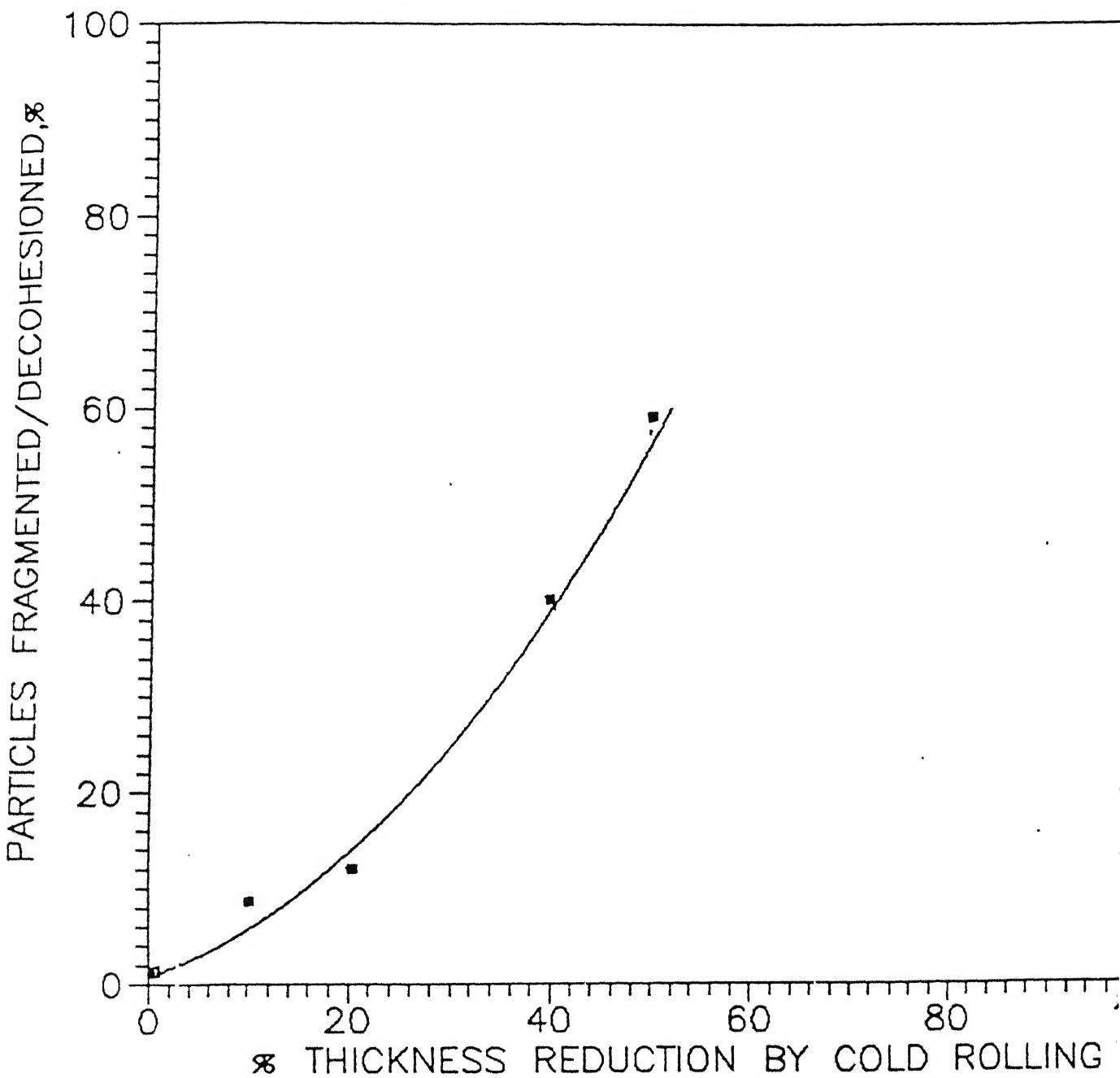


FIG 4.22 EFFECT OF COLD ROLLING THICKNESS REDUCTION
ON DECOHESION / FRAGMENTATION OF INCLUSIONS.

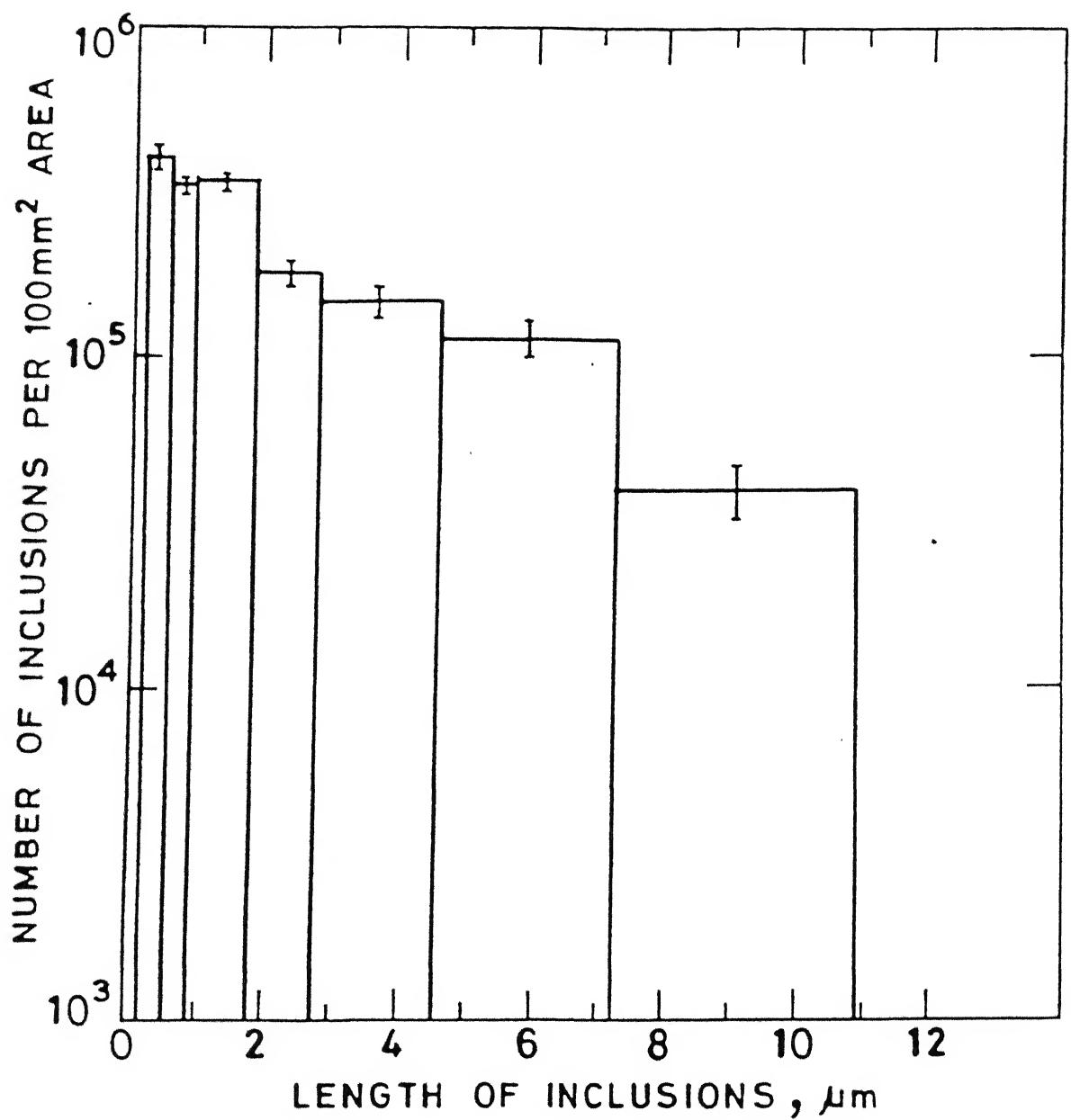


FIG.4.23 INCLUSIONS SIZE DISTRIBUTION HISTOGRAM FOR HR DIRECT COPPER STRIP.

behaviour of Cu_2O inclusion, copper strips made directly from cuprous oxide preform were cold rolled to 34% to 58% thickness reduction and observed under SEM. Fig. 4.24 show decohesion and/or Fragmentation of Cu_2O inclusion in the above strip.

The variation of Tensile strength and elongation as a function of cold rolling thickness reduction are shown in Fig. 4.25 and 4.26. It can be seen that both strength and ductility increased upto a thickness deformation of 40%. Beyond this thickness reduction by cold rolling the properties start deteriorating.

Many workers⁽²⁹⁻³⁷⁾ studied the mechanism of void formation in a plastically deformed matrix. Two basic kinds of damage processes can take place depending on the values of matrix particle interface stiffness and the particle stiffness

- (a) Decohesion at the inclusion-matrix interface and
- (b) Fragmentation of the inclusions

According to Schmitt and Jaliner⁽³³⁾ the following metallurgical and mechanical parameters make a sheet material very sensitive to damage around inclusion during its cold rolling.

- (a) the shape, size, distribution, chemical nature and plasticity of inclusions.
- (b) the cohesion between inclusion and matrix.
- (c) the inclusion orientation with respect to the principal axes of stress and strain.
- (d) the plastic behaviour of the matrix including its strain

behaviour of Cu_2O inclusion, copper strips made directly from cuprous oxide preform were cold rolled to 34% to 58% thickness reduction and observed under SEM. Fig. 4.24 show decohesion and/or Fragmentation of Cu_2O inclusion in the above strip.

The variation of Tensile strength and elongation as a function of cold rolling thickness reduction are shown in Fig. 4.25 and 4.26. It can be seen that both strength and ductility increased upto a thickness deformation of 40%. Beyond this thickness reduction by cold rolling the properties start deteriorating.

Many workers⁽²⁹⁻³⁷⁾ studied the mechanism of void formation in a plastically deformed matrix. Two basic kinds of damage processes can take place depending on the values of matrix particle interface stiffness and the particle stiffness

- (a) Decohesion at the inclusion-matrix interface and
- (b) Fragmentation of the inclusions

According to Schmitt and Jaliner⁽³³⁾ the following metallurgical and mechanical parameters make a sheet material very sensitive to damage around inclusion during its cold rolling.

- (a) the shape, size, distribution, chemical nature and plasticity of inclusions,
- (b) the cohesion between inclusion and matrix,
- (c) the inclusion orientation with respect to the principal axes of stress and strain,
- (d) the plastic behaviour of the matrix including its strain

behaviour of Cu_2O inclusion, copper strips made directly from cuprous oxide preform were cold rolled to 34% to 58% thickness reduction and observed under SEM. Fig. 4.24 show decohesion and/or Fragmentation of Cu_2O inclusion in the above strip.

The variation of Tensile strength and elongation as a function of cold rolling thickness reduction are shown in Fig. 4.25 and 4.26. It can be seen that both strength and ductility increased upto a thickness deformation of 40%. Beyond this thickness reduction by cold rolling the properties start deteriorating.

Many workers⁽²⁹⁻³⁷⁾ studied the mechanism of void formation in a plastically deformed matrix. Two basic kinds of damage processes can take place depending on the values of matrix particle interface stiffness and the particle stiffness

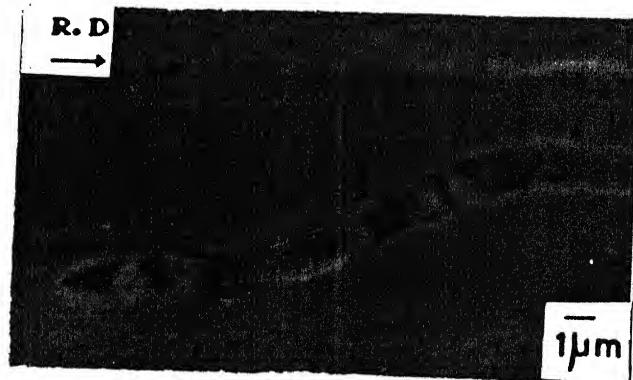
- (a) Decohesion at the inclusion-matrix interface and
- (b) Fragmentation of the inclusions

According to Schmitt and Jaliner⁽³³⁾ the following metallurgical and mechanical parameters make a sheet material very sensitive to damage around inclusion during its cold rolling.

- (a) the shape, size, distribution, chemical nature and plasticity of inclusions.
- (b) the cohesion between inclusion and matrix.
- (c) the inclusion orientation with respect to the principal axes of stress and strain.
- (d) the plastic behaviour of the matrix including its strain



a 34% CR (X5000)



b 58% CR (X5000)

Fig. 4.24 SEM views of fully dense copper strip, prepared from cuprous oxide powder cold rolled to different deformations, showing decohesion and/or fragmentation of inclusions.

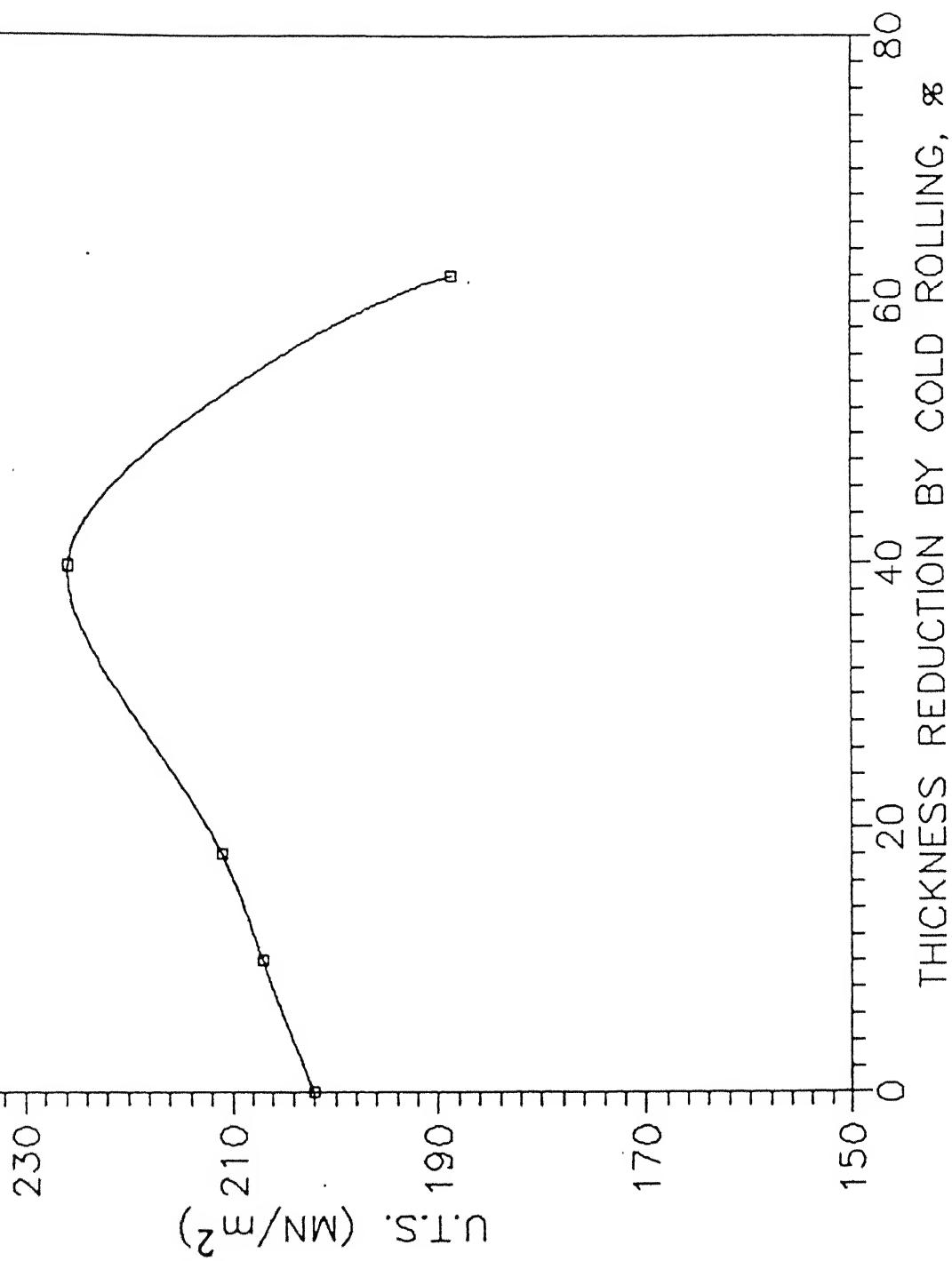
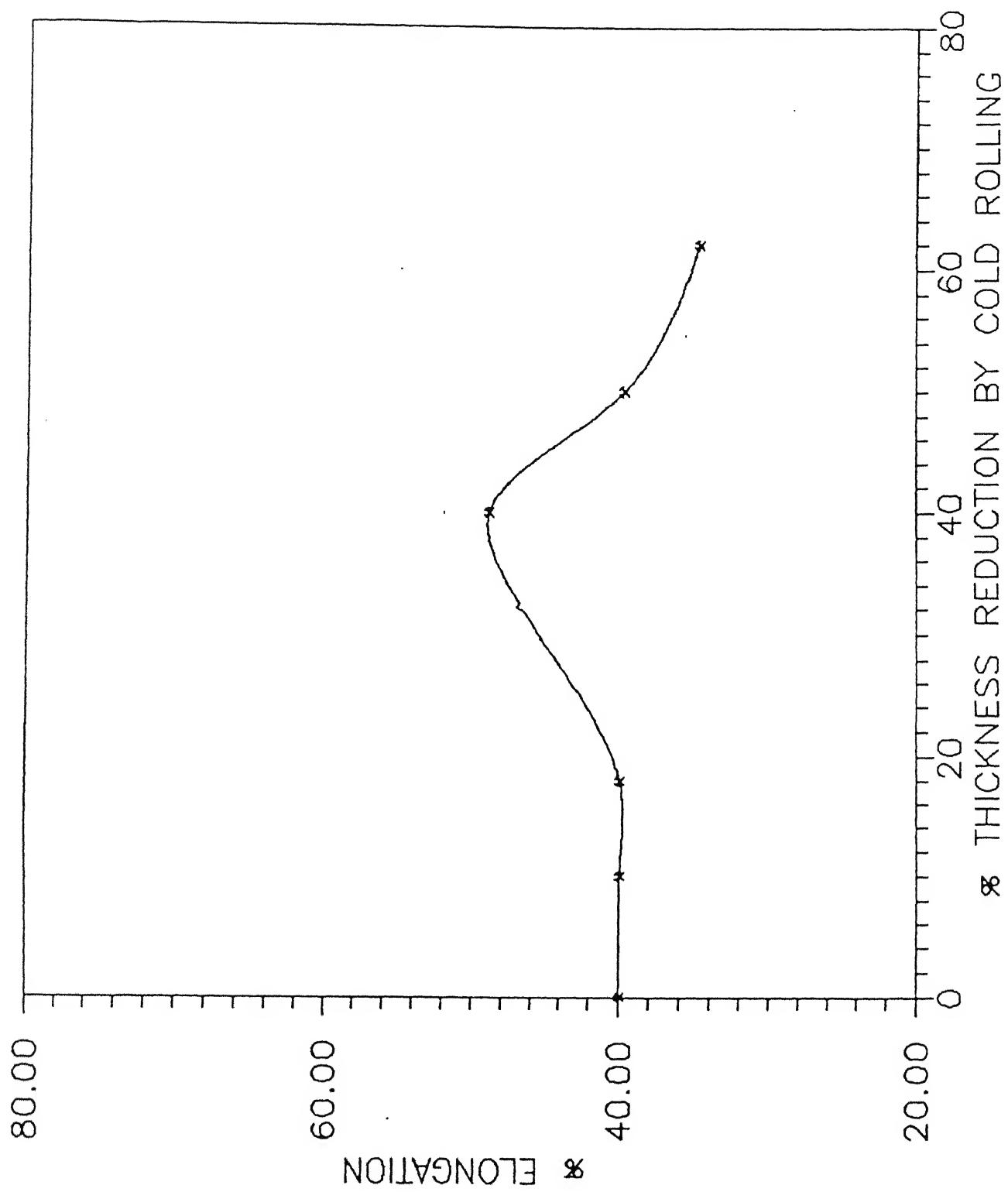


FIG 4.25 . EFFECT OF COLD ROLLING THICKNESS REDUCTION ON U.T.S. OF A STRIP HOT ROLLED & ANNEALED.



hardening characteristics and the strain rate sensitivity.

Tanaka et al⁽³⁴⁾ have studied the effect of size of inclusion on the cavity formation at interface of an inclusion, considering the energy criterion, they have formulated the value of critical strain required for the formation of cavity around a spherical inclusion in case of uniaxial tensile deformation. According to them the critical strain to cause cavitation is given by following equations -

$$\epsilon_{\text{crit}} = \left(\frac{1}{d} \right)^{1/2} \quad \text{when } \alpha < 1$$

$$\epsilon_{\text{crit}} = \left(\frac{1}{\alpha d} \right) \quad \text{when } \alpha \geq 1$$

where α is the ratio of youngs modulus of inclusion to that of the matrix and d is the diameter of the inclusion.

From the above equation it can be inferred that the strain required for cavitation is inversely proportional to the diameter of inclusion. A similar type of behaviour was observed in the present case as discussed earlier.

The nature of variation in the mechanical properties of strip as a function of thickness reduction can be discussed in the light of decohesion and/or fragmentation. As shown in Figs. 4.25 and 4.26 there is an increase in Tensile strength and Elongation upto 40% cold rolling. As discussed earlier, decohesion and/or Fragmentation of Cu_2O inclusion takes place during cold rolling of copper strip. Although this behaviour starts from the very

beginning of the cold rolling, there were only a few inclusion sites where decohesion and/or fragmentation has taken place upto 40% thickness reduction. The size of inclusions where such phenomenon has taken place is about $10 \mu\text{m}$. Therefore the main reason for the increase in the mechanical properties of cold rolled and annealed strip upto 40% thickness reduction seems to be due to grain refinement. The grain sizes of strips cold rolled to 10, 20, & 40 percent are given below:

As hot rolled $\rightarrow 12.18 \mu\text{m}$

18% Cold rolled $\rightarrow 10.88 \mu\text{m}$

40% Cold rolled $\rightarrow 8.65 \mu\text{m}$

The grain size decreased as cold rolling thickness reduction is increased. This shows that grain refinement has taken place. After 40% thickness reduction the properties decreased due to the increase in decohesion and/or Fragmentation of inclusions present in the copper strips as discussed earlier.

5. CONCLUSIONS

1. Sintered copper strips, containing about 65% porosity, made from atomized powder by the slurry casting method can sustain large amount of thickness reductions (upto ~ 80%) without any significant edge cracking by hot rolling in a single pass at all the temperature investigated in the present study viz 923K, 1023K, 1123K and 1223K.
2. Results of strip densification with the percentage thickness reduction imparted at 1023K and 1123K respectively show that the densification behaviour of the strip remains unaffected by the rolling temperature. Densification of porous strip at both the temperatures can be adequately described by the relationship.

$$e_t = 1 - \left[\left(\frac{\rho_0}{\rho} \right)^{\frac{C+3}{3}} \left(\frac{1-\rho^2}{1-\rho_0^2} \right)^{C/6} \right]$$

where, c is a constant having a value of 0.4 and is the ratio of youngs Modulus of the material to its shear Modulus. ρ_0 is the relative density of starting porous copper strip and ρ is the relative density of hot rolled strip at a given Fractional thickness deformation e_t .

3. A thickness reduction of about 80% imparted to porous copper strips of relative density of 0.35 by hot rolling them in a single pass produces fully dense strips.
4. Densification of porous copper strip upto a relative density of 0.7 involves the sliding of copper particles around their

neck negious leading to their transitional restacking. After the relative density of 0.7 increasingly larger particles deform by their longitudinal flow in the rolling direction and from inter particle contact areas with their co-ordinating neighbours. During this stage, the interconnected porosity starts transforming into isolated porosity. Eventually the collapsing of isolated pores occurs resulting into the full densification of the strip.

5. Optical micrographs show an elongated grain structure in strips hot rolled at all temperatues viz 923K, 1023K, 1123K and 1223K. However, the Aspect ratio is different at different temperatures. There is a decrease in Aspect ratio of grains as hot rolling temperature increased. Same nature observed in strips hot rolled at above temperatures and cooled in graphite chips.

Transmission electron micrographs of strips hot rolled at different temperatues show cell structure. This implies high dislocation density is present in the strips.

6. Fractured surfaces of Tensile samples of strips hot rolled at different temperatures show intergranular Fracture at low hot rolling temperatures and a partially dimpled structure at high hot rolling temperatures. The bond strength of particles at high hot rolling temperature is more compared to that at low temperatues.
7. The increase in Tensile strength, Elongation and yield strength is explained in terms of above microstructures and

Fractographs.

8. Strips which were produced from atomized copper powder contain fine Cu_2O inclusions. Size distribution analysis of inclusions obtained by image Analyser shows positively skewed, i.e. most of the inclusions were in the micron and submicron range with relatively few inclusions present in maximum size range of 10-14 μm .
9. Scanning Electron micrographs show that upto 40% cold rolling deformation the decohesion and/or Fragmentation has taken place at only few inclusion sites. The size of inclusion where such phenomenon takes place is about 10 μm . Above 40% thickness reduction the decohesion and/or Fragmentation increased as the thickness reduction by cold rolling increased.
10. Tensile properties obtained on cold rolled strips and annealed at 823°K for 1800 sec under Argon atmosphere show that Tensile strength, Elongation increase upto 40% and deteriorate in strips which are cold rolled beyond 40% thickness reduction. The nature of this behaviour is explained in light of the SEM micrographs.

CHAPTER - 6

SUGGESTIONS FOR FURTHER STUDY

- (1) In the present investigation, the study on transitional restacking, development of contact areas and longitudinal flow of particles has been only on a qualitative basis. This can be studied quantitatively using fundamental principles of quantitative metallography.
- (2) Results of this study show that deterioration of mechanical properties occur due to decohesion and/or fragmentation of inclusions. Efforts should be made to correlate different processing parameters with observed inclusion size distribution in hot rolled and annealed strips.
- (3) Cuprous oxide inclusions can be present in strips made from conventional methods. Therefore during cold rolling there will be decohesion and/or fragmentation of inclusions as studied by earlier workers. Systematic studies can be done on strips made from conventional methods, so as to compare with P/M metal strips.

REFERENCES

1. Marsden, T.B. and Hall, A.V.; Metals Technology 2(3), 1975, 98.
2. Biswas, A.K. and Davenport, W.G.; Extractive metallurgy of copper; Permagon Press, Oxford, 1976.
3. Dowsing, R.J. ; Metals and Materials, 15 (5,6), 1981, 62.
4. Dube, R.K.; Powder Met. Int., 13(4), 1981, 188.
5. Florance, J.R., BNF conference on 'Maximising the yield of nonferrous metals, Liege, 1971, 118.
6. Hill, D.A., Metals Technology, 2(3), 1975, 130.
7. Chapman, P.F., Metals and materials, 8(2), 1974, 107.
8. RO. D.H. and Toaz, Progress in powder metallurgy 1982; vol 38 eds. Bewley, J.G. and McGee, S.W.; APMI. Princeton, 311.
9. Diebel,C, Thomburg, D.R. and Emley, F.; Powder Met., 3(5), 1960, 32.
10. Dube, R.K., International Materials Review, 35 (5) 1990, 253.
11. Reen, D.W.; Progress in powder metallurgy; vol 20, MPIF N.Y., 1964, 194.
12. Weaver, C.H., Butters, R.G. and Lund, J.A. "Int. J. Powder Met & Powder Tech; 8(1), 1972, 3.
13. Blore, M.H.D.; Sillins, V.; Romanchuk, S.; Benz, T.W. and Kackiw, V.N.; Metals Eng. Quan, 6(2), 1966, 54.
14. Smucker, R.A.; Iron and Steel Engg. 36(2), 1959, 128.
15. Williams, J.C.; 'Treatise on Material Science and Technology'. Vol 9, Academic Press, N.Y., 1976, 173.

16. Onada, G.Y., Jr.; 'Ceramic processing before firing', ed Onada G.Y. and Herich, L.L. John Wiley & Sons, Inc., N.Y., 1978, 235.
17. Misfler R.E. & Shanefield, D.J.; 'Amer. ceramic society bulletin' 53(5), 1974, 416.
18. Thompson, J.J.; Amer. ceramic society bulletin, 42(9) 1963, 480.
19. Arthur, G.; J. Inst. Metals 83, 1954-55, 329.
20. Tension Testing of Metallic Materials, E8-61T ASTM standards, Part 3, American society of testing materials, Philadelphia, 1961, 165.
21. KEHL, Principles of Metallographic Laboratory Practice.
22. Cockayne; Phil. Mag. 24, 1971, 1383.
23. Dewey, M.A.P. & Lewis, T.G., J. Sci. Instrum. 40, 1963, 385.
24. Estimating the average Grain size of Wrought copper and copper base alloys', E79 - 49T, ASTM standards, Part 3, American society for Testing materials, Philadelphia, 1961, 625.
25. Bhargava, S.; Met. Trans in Press.
26. Artz, E. and Fisenmeister, H.F.; Powder Metallurgy 26(2), 1983, 82.
27. Jonas, J.J. & McQueen, H.J., Treatise on Material Sci. and Tech. Vol 7, Academic Press, N.Y., 400.
28. Katrus, D.A.; Sov. Powder Met. and Metal ceramics, 163(7), 1976, 61.
29. Baker, C. and Smith, G.C.; Trans. Met. Soc. AIME, 242, 1968, 1989.
30. Goods, S.H. and Brown, L.M.; Acta Metall., 27, 1979, 1.

31. Roberts Williams and Kenneth, E. Easterling; *Acta Metall.*, 24, 1976, 745.
32. Schmitt, J.H., Jalinier, J.M., and Baudelet, B.; *J. Mater. Sci.* 16, 1987, 95.
33. Schmitt, J.H. and Jalinier, J.M.; *Acta Metall.* 30, 1982, 1989.
34. Tanaka, K., Mori, T. and Nakamura, T.; *Phil. Mag.* 21, 1970, 267.
35. Fischmaster, H.F., Navara, E. and Easterling, K.E., *Metall. Sci. J.* 6, 1972, 211.
36. Argon, A.S. and Im, J.; *Met. Trans.*, 6A, 1975, 839.
37. Jalinier et al. *J. Mat. Sc.* 16, 1981, 2004.
38. Dube R.K. and Bhargava, S., *Mater. Sci. Technol.*, 1985, 1(9), 743.

A112178

ME-1991-M-RAM-STU